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1ST AND 2ND CROERS													PROCESS AND PREPARED BY													3RD AND 4TH CROERS									
<p><i>24</i></p> <p>Nitration products of starch. 1. Preparation of starch nitrates from potato starch. JULIUS HACHEL AND TADEUSZ URBANSKI. <i>Roczniki Chem.</i> 12, 270-97 (in French 294-7) (1932).—In nitrating potato starch with HNO_3, the following conclusions are drawn: An increase of the acid concn. causes an increase of the N content and of the viscosity of the nitrates. Stabilizing boiling of the nitrates has a detrimental effect inducing denaturation and an increase of the poly. in EtOH. The N content increases with increasing amt. of HNO_3 used. A rise of the temp. from 0° causes a lowering of the N content, of the yield and of the viscosity of the nitrates, accompanied by an increase of the EtOH soly. The changes are probably due to secondary oxidation reactions induced by the rise in temp. With increasing time of esterification the N content of the product rises rapidly at first, and then the increase becomes steadily smaller. The increase of the nitration period favors also the above secondary reactions, causing a drop of the yield, of the N content and of the viscosity of the product. Starch nitrates pptd. by H_2O show a better chem. stability than those pptd. by H_2SO_4. In nitrating starch with $\text{HNO}_3\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$ mixts. the best yield is obtained with mixts. containing equimolar amts. of H_2SO_4 and H_2O. Such mixts. showing a max. partial pressure of HNO_3 vapors exhibit a max. nitrifying power, and behave here in the same manner as in the nitration of cellulose. Mixts. poor in H_2SO_4 differ in their effect on starch from that on cellulose insofar as nitrated starch is probably sol. in them.</p> <p style="text-align: right;">J. WIENBLAK</p>																																			
<p>ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																																			

ca
Photographic studies of detonation of explosives. Tadeusz Urbanski. *Rozprawy Chem.* 12, 715-89 (768-9 in French) (1932); cf. *C. A.* 27, 4930.—The detonation process of *picric acid* and *nitrocellulose* was studied by means of a high-speed camera permitting the dissection of a detonation flame into a series of pictures differing from each other by a distance of $1/1000$ of a sec. With *picric acid* the primary flame represents the main and partly the secondary reaction and consists of a center surrounded by luminous bundles. The progress of the secondary reaction depends on the rate of cooling of the reaction products effected by reduction of the pressure. The luminescence is due to glowing of free C and is induced by various reactions in the explosion space. The photographic record permits the establishment of the following scheme of the explosion progress: The charge, being the center of the primary flame, is the place of the decomn. of the explosive. The decomn. products interact immediately, forming brighter reaction zones upon a darker field. This state is especially well perceived in photographing blue rays, in case the explosive is confined only with celluloid or is entirely free. If it is placed in a glass tube, the different zones run into each other causing brightening of the whole field, upon which darker bands are visible. The interacting decomn. products diverge and form luminous bands around the center. The glowing ceases with time toward the inner part whereby rings

24
around the center are formed. The diam. of the rings increases steadily until the light is extinguished. The space between the center and the rings is illuminated by numerous radiant points. Analysis of the primary flame permits the conclusion that the reactions of the primary decomn. products do not proceed evenly in the whole

mass, but are broken up into secondary centers forming rings around the place of the detonation. The flame along the charge axis shows all characteristics of an explosion flame. After extinction of the primary flame, a secondary one appears induced by oxidation of the reaction products: C, CO, CH₄, H₂, HCN, etc. The primary flame contains all colors of the visible spectrum; in the secondary one, blue is observed feebly, red and infra-red intensely. The lowest temp. limit of the primary flame is calcd. to 2000-2300°. Nitrocellulose behaves similarly, except that the form of the rings around the center is more regular, and their diam. is smaller than with *picric acid*. The lowest temp. of nitrocellulose, having 13.3% N, is calcd. to 1900-2000°. J. W.

ASB-35A METALLURGICAL LITERATURE CLASSIFICATION

PROCESS AND PROPERTY INDEX

H-1

Phenomena associated with detonation of explosives. T. Ullrich (Russ. Chem. 1908, 12, 126—127).—Mixtures of H_2O_2 with $\text{C}_2\text{H}_5\text{NO}_2$, or with glyceryl nitrate yield on detonation in glass tubes rapidly propagating explosion flames. The velocity of propagation of the flame varies at different parts of the tube at the same distance from the origin. These effects are ascribed to asymmetrical action of the detonator, and to heterogeneity of the medium. A thin layer of explosive in contact with the walls of the tube may explode after the central mass, or may even remain intact after the explosion.

R. T.

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

FROM STUDYING

FROM DONATING

LARGES +A

SMALLER +B

EXPLANATION

REMARKS OR OTHER DATA

24

Chemical Abstracts

Nitration products of starch. II. Preparation of starch nitrates from starch from different sources. JULIUS HACKEL AND TADOMIR URBANSKY. *Russkii Khim* 13, 221 A(225 in French (1933). cf. C. I. 27, 2092. No marked differences were found in the N content of nitrates prepared from corn, rice, tapioca or potato starch, or from sol starch, in contradiction of the findings of Berl and Butler (C. I. 4, 1550).

T. H. CHILTON

ASD-514 METALLOGRAPHIC LITERATURE CLASSIFICATION

Thermal analysis of binary mixtures containing organic nitrates. Indurum Urquiza, *Rennell Chem.* 13, 309-330 (1933). A 7/8-in. sample, in a 12-mm. tube placed in a 16-mm. tube which in turn is placed in a 35-mm. tube coated with glycerol, is heated to 5-10° above its m. p. During the cooling, the melt and glycerol are stirred vigorously. Temp.-comp. curves are given. The following figures are, resp., the percentage of the 1st ingredient and the eutectic pt. Mannitol nitrate- β -nitrotoluene, 12.5, 50.2°; β -nitroanisole 8, 50.8°; β -nitrophenetole, 5, 50.5°; α -chloronitrobenzene, 18.5, 39.7°; α -nitroanisole, 14, 52.5°; α -dinitrobenzene, 32.5, 63.5°; 1,3-dinitroanisole, 55, 77.8°; 1,2,4-chlorodinitrobenzene, 25, 44.5°; γ -trinitrobenzene, 55, 78.7°; α -trinitrophenol, 42.5, 63.5°; naphthalene, 30, 77.2°; dimethylphosphorylurea, 57, 77.4°; diethylphosphorylurea, 53-4, approx. 82.1°. Nitroerythritol- β -nitrotoluene, 47, 32.4°; α -dinitrobenzene, 70, 42.4°. Pentaerythritol nitrate- β -nitrotoluene, 10, 50.5°; β -nitroanisole, 5, 50.8°; α -nitronaphthalene, 4, 55.1°; α -dinitrobenzene, 20, 53.4°; 1,2,3-dinitroanisole, 20,

94.7°: -1,2,4-chlorodinitrobenzene, 3, 40°: -sym-trinitrobenzene, approx. 30, 101.1°: -*a*-trinitrotoluene, approx. 13, 76.1°: -naphthalene, approx. 7, 79.1°: -dimethyldiphenylurea, 32.5, 102.7°: -diethyldiphenylurea, approx. 12, 68°: mannitol nitrate-erythritol nitrate 18.5, 57.6°: pentaerythritol nitrate-mannitol nitrate, 20, 101.3°: pentaerythritol nitrate-erythritol nitrate, approx. 5, 56.5°. Mannitol nitrate was the only compd. giving rise to mol. addn. compds. Probable compds. (dett. by extrapolation) are, in mols. per mol. of mannitol nitrate, *p*-nitrophenol 2, *p*-nitroanisole 2, *p*-nitrophenetole 2, *a*-nitronaphthalene 1. Bibliography. C. T. I.

ASME-314 METALLURGICAL LITERATURE CLASSIFICATION

The solubility of triisro-trimethylene-trinitramine.
Tadeusz Urbanski and Boleslaw Kwiatkowski. *Roczniki
Chem.* 18:~~690~~-7247 in French(1933).—The soly. of
hexogen in the following solvents Me₂CO, MeOH, EtOH,
Et₂O, iso-AmOH, CCl₄, PhCH₃, and CCl₄ was detd.
The ease of soly. decreases in the order of solvents given
above. C. T. Ichulowski

ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION

[illegible]

131 APR 270 0800

PROCESSING AND PH-0000000000

100 AND 01000000

COUNCIL ELEMENTS

BC

Thermal analysis of mixtures containing tri-
nitrophenylhydrazones: T. Ohsaki and I.
Kawanabe (Repts. Chem. Ind. Jpn. 1964; 1965).—Neither solutions nor compounds are formed
in the system: tri-nitrophenylhydrazones—
p-C₆H₄NO₂—NO₂—C₆H₄NO₂—1-C₆H₄NO₂
—m-C₆H₄NO₂—Cl—NO₂—C₆H₄NO₂—C₆H₄NO₂
—Cl—NO₂—OH, m-C₆H₄NO₂—OH, NPA—CO—NH₂,
NPA—CO—NH₂, oxalophen, and NHPh. R.T.

131 APR 270 0800

PROCESSING AND PH-0000000000

100 AND 01000000

COUNCIL ELEMENTS

BC

Thermal analysis of mixtures containing tri-
nitrophenylhydrazones: T. Ohsaki and I.
Kawanabe (Repts. Chem. Ind. Jpn. 1964; 1965).—Neither solutions nor compounds are formed
in the system: tri-nitrophenylhydrazones—
p-C₆H₄NO₂—NO₂—C₆H₄NO₂—1-C₆H₄NO₂
—m-C₆H₄NO₂—Cl—NO₂—C₆H₄NO₂—C₆H₄NO₂
—Cl—NO₂—OH, m-C₆H₄NO₂—OH, NPA—CO—NH₂,
NPA—CO—NH₂, oxalophen, and NHPh. R.T.

2

CO

Thermal analysis of binary mixtures containing organic nitrates. T. Urbanek. *Rozprawy Chem.* 16, 925-40 (1934); cf. C. A. 28, 279. The m-p. diagrams of the systems mannitol hexanitrate (I)-p-NO₂C₆H₄OH (II), p-C₆H₄NO₂, p-C₆H₄(OMe), m-(III) and p-(IV)NO₂, C₆H₄CO₂H, m-(V) and p-NO₂C₆H₄CHO, m-NH₂C₆H₄NO₂, and 1,2,4-C₆H₃Me(NO₂)₃ (VI); erythritol tetranitrate (VII)-(IX) and 2,4,6-trinitrotoluene (VIII); pentacerythritol hexanitrate (X)-(VI), trinitrophenylmethylnitrothiolate (X)-(VI), and camphor (X), indicate the unstable compounds. In III, IV, V, VI, VII, VIII, and IX, stable compounds are formed in the system IX X, while in the remaining systems only ordinary eutectic mixts. are observed. B. C. A.

CLASSIFICATION

ASB-SEA METALLURGICAL LITERATURE

COMMON ELEMENTS										PROCESSES AND PROPERTIES										MATERIALS									
COMMON ELEMENTS										PROCESSES AND PROPERTIES										MATERIALS									
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<p>Thermal analysis of the system picric acid-dinitro- methylolane. T. Urbaniski and B. Kwiatkowski. <i>Roczn. Chem.</i> 10, 941-8(1984).--Neither 1,5- nor 1,8- $C_6H_3(NO_2)_3$ gives rise to compds. or solid solns. B. C. A.</p>																													
<p>ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																													

BC

Solubility of cellulose nitrate. T. UHRAHAKI
(Rec. Chem., 1934, 24, 944-956).—The solubility
 S , of cellulose nitrate (I) in mixtures of EtOH and
various solvents (Me, Et, Pr, Pr^i , Bu, Bu^i , and
iso-C₄H₉), acetone and benzene, EtOOEt, PrOOEt,
EtOOEt, CH_2Cl_2 , CH_2Br_2 , CO_2 , and CO_2 (liq.)
is expressed by $S = k \cdot R^x$, where R is the % of (I)
dissolved by the solvents, and x is the concn. of
solvent. For the series EtOOEt, S increases with
increasing mol. wt. of Et, and the max. time, the
dielectric const. ϵ of the solvent diminishes, and the
dipole moment μ increases, so that S varies parallel
with μ/ϵ . Analogous relationships are not found
between the val. of S and the mol. wt. of R.

R. T.

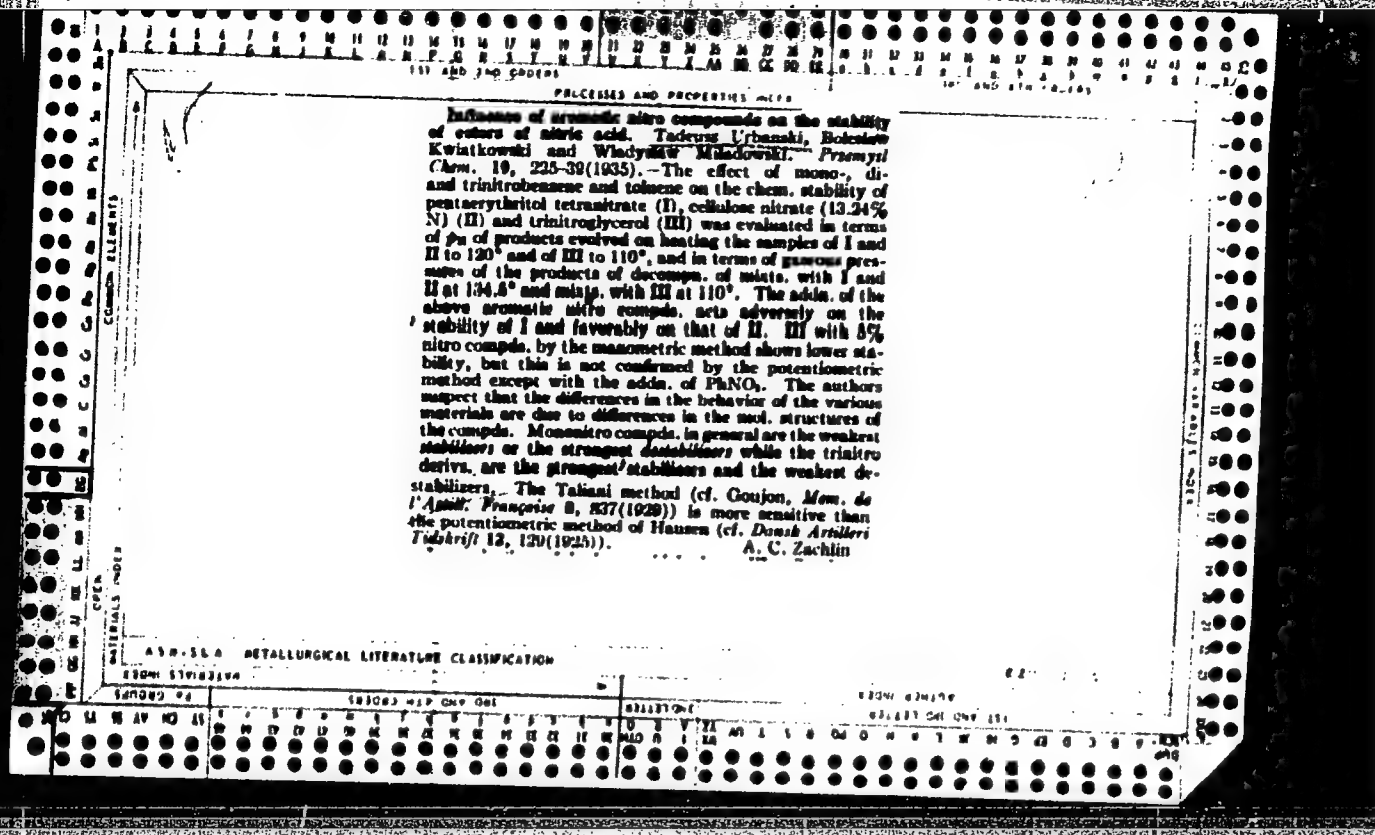
EXPLOSIVE PROPERTIES OF STARCH NITRATES. J. HACHET and T. UMANOWSKI (Framm's Chem., 1884, 10, 398-401).—A study of the explosive properties (I) of starch nitrates (II), as expressed by the velocity of detonation, the Pb block test, brisance, and the sensitivity to shock; indicates that (I) augment with the N content, and that (II) containing < 9% N have no practical val. as explosives, whilst the (I) of (II) containing > 9% N are comparable with those of $C_6H_5Me(NO_2)_3$ and $HO-C_6H_4(NO_2)_3$. R. T.

157 AND 158										159 AND 160									
PROCESSING AND PREPARATION																			
<p>Thermal analysis of binary mixtures containing organic nitrates. III. T. Urbanek. <i>Repts. (Arm. 13, 191-7 1955); cf. C. A. 50, 6129.</i>—Temp.-comp. curves of the systems studied are given. The accompanying figures are resp. the percentage of the first ingredient and the eutectic point. Mannitol nitrate-<i>m</i>-nitrotoluene forms an addn. compd. (in ratio of 1:2) which forms a eutectic with mannitol nitrate, 8, 14.4°; mannitol nitrate-sulfur, 45, 5.8°. Erythritol nitrate-1,2,4-dinitrotoluene, 61, 40.1°; -<i>sym</i>-trinitrobenzene, 67, 45.8°; -diethyl-phenylurea, 37, 42.2°; erythritol pentanitrate-phthalic anhydride, 20, 65.9°; -<i>Et</i> phenylcarbamate, 1, 48.6°; erythritol nitrate-1,2,4,6-tetranitrobenzene forms an addn. compd. (1 + 2) and yields 2 eutectics: one with the nitroaniline and the other with erythritol nitrate m. at 58.6° and 52° and corresponding to 10 and 70% of erythritol nitrate, resp.</p> <p>C. T. Ichikawa</p>																			
<p>ASB-55A METALLURGICAL LITERATURE CLASSIFICATION</p>																			
<p>157 AND 158</p>										<p>159 AND 160</p>									

ca

X-ray studies of nitrated starches. I. Maria Kolacz-
kowska and Tadeusz Urbaniski. *Roczniki Chem.* 15,
339-42(1935). The authors obtained x-ray spectrograms
of nitrated potato and sol. starch by the Debye-Scherrer
method. Both show a marked resemblance and give the

ASB SLA METALLURGICAL LITERATURE CLASSIFICATION



137 AND 138 CORDS										PROCESS AND PROPERTIES INDEX										137 AND 138 CORDS									
<div style="position: absolute; top: 10px; left: 10px; font-size: 2em; font-weight: bold;">BC</div> <div style="position: absolute; top: 10px; right: 10px; font-size: 1.5em;">2-1</div> <div style="position: absolute; top: 300px; left: 250px; text-align: center;"> <p>Thermal analysis of mixtures of ammonium nitrate, guanidine nitrate, and nitroguanidine. T. Uppelmann and J. Sauerbrey (Ber. Chem., 1900, 33, 285-288). — Data for the binary eutectics are: NH₄NO₃ (I) 74, guanidine nitrate (II) 26%, f.p. 127.7°; (I) 86, nitroguanidine (III) 20%, f.p. 131.5°; (II) 80, (III) 41%, f.p. 160.5°; those for the ternary eutectic are: (I) 69, (II) 23.5, (III) 17.5%, f.p. 112.2° R. T.</p> </div>										<div style="position: absolute; top: 370px; right: 10px; transform: rotate(90deg); font-size: 0.8em;">COMMON VARIABLE INDEX</div>																			
<div style="display: flex; justify-content: space-between;"> <div> <p>COMMON ELEMENTS</p> <p>COMMON VARIABLE INDEX</p> <p>COMMON VARIABLE INDEX</p> </div> <div> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> </div> </div>																													
<p>ASB-55A METALLURGICAL LITERATURE CLASSIFICATION</p>																													
<div style="display: flex; justify-content: space-between;"> <div> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> </div> <div> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> <p>137 AND 138 CORDS</p> </div> </div>																													

1ST AND 2ND ORDERS										3RD AND 4TH ORDERS									
PROCESSING AND PROPERTY INDEX																			
<p>BC</p> <p style="text-align: right;">2-1</p> <p>Theoretical analysis of binary mixtures containing esters of nitric acid. IV. T. URAKAWA (Rec. Chem., 1933, 10, 305-306).—The data suggest formation of unstable 1:2 compounds of /anionol hexanitrate (I) with PhNO_2, $\text{c-C}_6\text{H}_4\text{NO}_2$, and Et α-nitroacetate, and of a 2:1 compound with Et α-nitroacetate. Stable esters are formed in the systems: (I)-$\text{PhOCH}_2\text{CH}_2\text{NO}_2$, erythritol tetranitrate $\text{c-OEt-C}_6\text{H}_4(\text{NO}_2)_2$, and penterythritol tetranitrate $\text{m-NO}_2\text{C}_6\text{H}_4\text{NH}_2$ and $\text{m-C}_6\text{H}_4\text{O}_2$. R. T.</p>																			
<p>ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																			
<p>1ST AND 2ND ORDERS</p>																			
<p>3RD AND 4TH ORDERS</p>																			

1ST AND 2ND ORDERS										3RD AND 4TH ORDERS									
PROCESSES AND PROPERTIES INDEX																			
<p>BC</p> <p>Thermal analysis of mixtures containing ammonium, sodium, potassium, and calcium nitrates. T. Umeda and S. Kozubinski (Recs. Chem., 1938, 22, 110-123).--The system $\text{NH}_4\text{NO}_3\text{-Ca(NO}_3)_2$ (eutectic, 71% of NH_4NO_3, 111') belongs to Rosenbloom's type V. Thermal diagrams for the systems $\text{NH}_4\text{NO}_3\text{-NaNO}_3\text{-KNO}_3$ and $\text{NH}_4\text{NO}_3\text{-NaNO}_3\text{-Ca(NO}_3)_2$ have been determined. R. T.</p>																			
<p>ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																			
FROM SYMBOL										FROM SYMBOL									
SYMBOL										SYMBOL									

Susceptibility of explosives to shock. I. Method.
Tadeusz Urbanaki. *Przemysl Chem.* 20, 117-27 (1936).
The literature is reviewed; 24 references are given. A modification of the methods of Lenze (*Atti VI Congr. Intern.* 2, 322 (1907)) and Kast (*C. A. S.* 2226) is adopted. Agreement between results can be improved if the following precautions are observed: The plunger of the app. should be subjected to more than 10 or 20 strokes before making the detna. In order to arrive at such a condition that the end of the plunger will not be further deformed by the blow. The harder the steel the fewer the blows needed to stabilize the plunger. U. shows clearly the effect of such stabilization. The rebound for each lot of plungers from various heights should be detd. for each lot of plungers similarly prepd. from the same material. From this the work done by the wt. is calcd. and expressed in kg./sq. cm. At least two app. should be used alternately. The quantity of substance used each time should be standardized at 0.01 to 0.02 g. The substance should be pulverized to a grain size of about 0.1 mm. diam. The app. should be cleaned after every stroke, and all deformations should be cleaned off sufficiently to describe the be-pothashed away. In order sufficiently to describe the behavior of the material under shock at least 2 heights should be detd. with an accuracy of 1.0 cm.; the min. at which 10% explosions are obtained and the max. for 50% of explosions. Each of the above should be detd. on the basis of at least 10 readings. In view of the varied be-

haviors of different explosives under shock at least 2 standard substances should be used, e. g., *hexogen* for the more sensitive substances and *picric acid* for the less sensitive substances. When the report of the explosion is questionable, then the appearance of soot or the presence of color of combustion should serve as addnl. criteria of explosion. An accuracy of 10% is obtained. II. **Sensibility of mixtures.** *Ibid.* 179-187. By the method described above, mixts. of *penthris* and *hexogen* with various nitro compds., an O carrier, and a neutral salt were studied. Mixts. of 2 explosive substances of different sensibilities show a sensibility which varies with compo., but the transition of mixts. from those rich in the sensitive component to those rich in the other shows a pronounced discontinuity. If a not very sensitive substance, e. g., *tetryl*, *tritol* or *picric acid*, is added to the extent of 10% to a sensitive substance such as *hexogen* or *penthris* the sensitiveness of the latter is increased. Sensitiveness is also increased by the addn. of an O carrier such as KNO_3 up to 80% or of a neutral salt (KCl). The addn. of a small quantity (5%) of a sensitive substance such as *hexogen* or *penthris* to such substances as nitro compds. increases the sensitiveness markedly. The above observations are interpreted to point to one conclusion: the increase in sensitiveness is produced by mech. sepn. of crystals of the foreign substance, which sepn. results in greater interior friction of the mass. A. C. Z.

ASB-3LA METALLURGICAL LITERATURE CLASSIFICATION

SUBJECT INDEX										AUTHOR INDEX									
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

1ST AND 2ND COLUMNS

PROCESSES AND PROPERTIES INDEX

2

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Henry Le Chatelier (1850-1936)—Creator of the theory of explosion. T. Uchibashi. *Kokushi Chem.* 17, 53 (1937).—Obituary notice. M. Wojciechowski

ASS-SLA METALLURGICAL LITERATURE CLASSIFICATION

SECTION 17

SECTION 18

SECTION 19

SECTION 20

SECTION 21

SECTION 22

SECTION 23

SECTION 24

SECTION 25

SECTION 26

SECTION 27

SECTION 28

SECTION 29

SECTION 30

SECTION 31

SECTION 32

SECTION 33

SECTION 34

SECTION 35

SECTION 36

SECTION 37

SECTION 38

SECTION 39

SECTION 40

SECTION 41

SECTION 42

SECTION 43

SECTION 44

SECTION 45

SECTION 46

SECTION 47

SECTION 48

SECTION 49

SECTION 50

SECTION 51

SECTION 52

SECTION 53

SECTION 54

SECTION 55

SECTION 56

SECTION 57

SECTION 58

SECTION 59

SECTION 60

SECTION 61

SECTION 62

SECTION 63

SECTION 64

SECTION 65

SECTION 66

SECTION 67

SECTION 68

SECTION 69

SECTION 70

SECTION 71

SECTION 72

SECTION 73

SECTION 74

SECTION 75

SECTION 76

SECTION 77

SECTION 78

SECTION 79

SECTION 80

SECTION 81

SECTION 82

SECTION 83

SECTION 84

SECTION 85

SECTION 86

SECTION 87

SECTION 88

SECTION 89

SECTION 90

SECTION 91

SECTION 92

SECTION 93

SECTION 94

SECTION 95

SECTION 96

SECTION 97

SECTION 98

SECTION 99

SECTION 100

1ST AND 2ND ORDER		PROCESSES AND PROPERTIES INDEX		100 AND 5TH ORDER	
<p><i>ca</i></p> <p>Nitration of paraffin hydrocarbons. II. - I. Urbanski and M. Sion. <i>Roczniki Chem.</i> 17, 101-4 (in French 194) (1947); cf. <i>C. A.</i> 31, 654¹.—Continuing the study of nitration of paraffin hydrocarbons with NO_2 in the gaseous phase, hydrocarbons from pentane to nonane were nitrated. The process was conducted at 200° and a mixt. of mono- and di-nitro compds. has been obtained with an efficiency of 30-80%, depending on the conditions of reaction. The formulas of the products are: $\text{C}_5\text{H}_{11}\text{NO}_2$, $\text{C}_6\text{H}_{13}\text{NO}_2$, $\text{C}_7\text{H}_{15}\text{NO}_2$, $\text{C}_8\text{H}_{17}\text{NO}_2$, $\text{C}_9\text{H}_{19}\text{NO}_2$. M. Wolczenkowski</p>					
<p>ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>					
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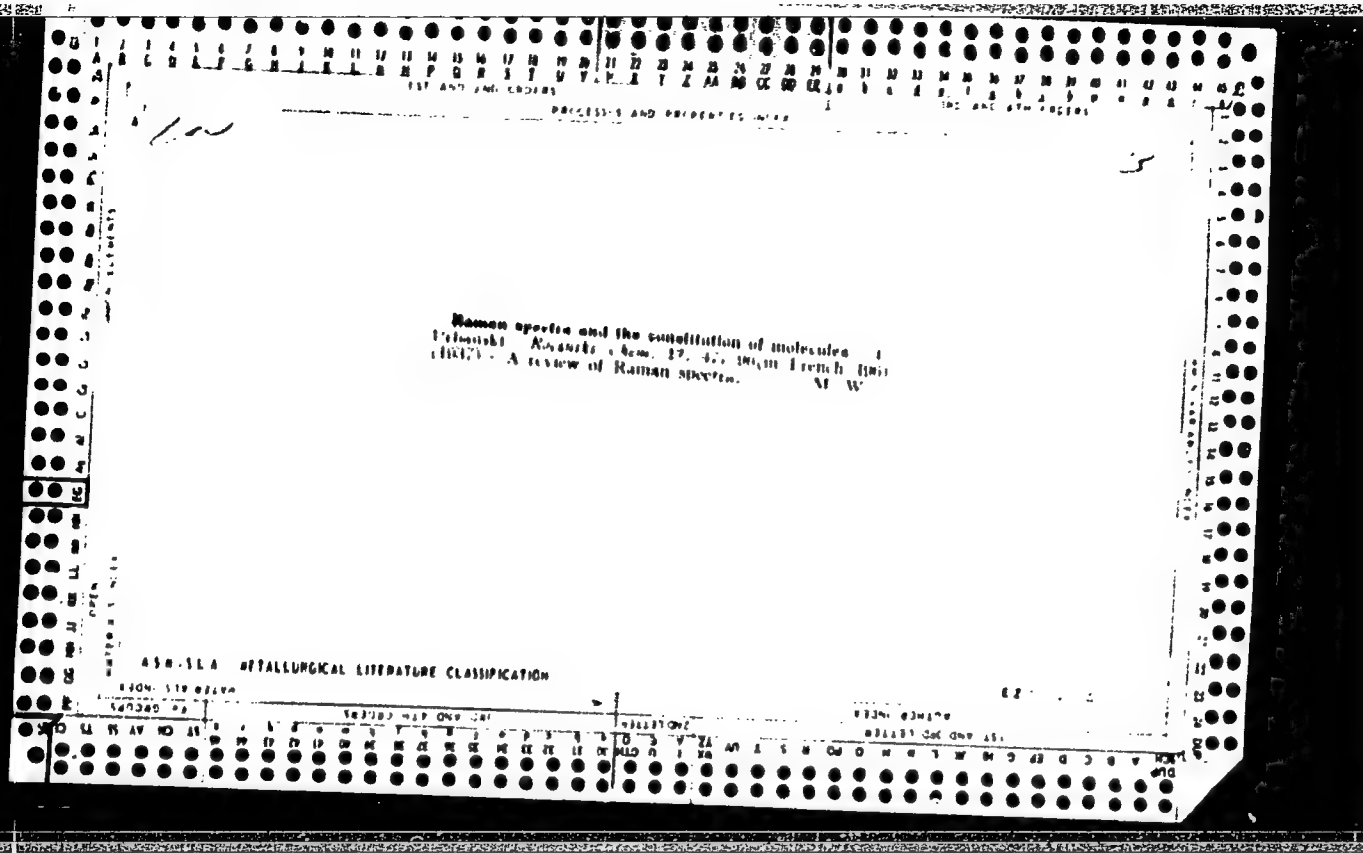
1ST AND 2ND CODES																										3RD AND 4TH CODES																									
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<p style="text-align: center;">24</p> <p style="text-align: center;">Nitration of cellulose and starch by means of nitric anhydride. T. Urbanicki and Z. Jankowski. <i>Rozprawy Chem.</i> 17, 349-52 (in French 200) (1957).—The authors have examined the nitrating action of N_2O_5 on cellulose and starch. By the action of N_2O_5 it is possible to obtain nitrocellulose contg. about 14% of N. The same result is obtained when liquid N_2O_5 mixed with HNO_3 acts on cellulose. Starch nitrated with gaseous or liquid N_2O_5 contains up to 13.86% of N. M. Wojciechowski</p>																																																			
<p style="text-align: center;">ASS-3LA METALLURGICAL LITERATURE CLASSIFICATION</p>																																																			
<p style="text-align: center;">1ST AND 2ND CODES</p>																																																			
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24

Calorimetric studies of thermal transformations of nitro-cellulose powders. W. Swietonowski, T. U. Gotski, H. Cahus and M. Rosidaki. *Rozwini Chem.* 17, 444-53 (in English 482-3) (1937).--A calorimeter devised for measuring very small heat effects evolved in processes of long duration was used. Old nitro-cellulose powder after preparation was used. Old nitro-cellulose powder, which slowly was heating to 75° showed a heat effect, which slowly disappeared; after a second heating to 75° the process of decomposition proceeded with increasing velocity. Freshly prepd. gun powder showed a very small heat effect, which disappeared after some time; a second exposure to the air caused its reappearance. Further contact with air or even with oxygen caused the heat effect to disappear.

M. Woickichowski

ASB-514 METALLURGICAL LITERATURE CLASSIFICATION



24

Preparation of trinitrotris(methylamino)benzene. T. Urbanaki. *Kocsiéti Chem.* 17, 501-3 (in French 502-3) (1937).—U. has prepd. 2,4,6-trinitro-1,3,5-tris(methylamino)benzene (I) by boiling *s*-trinitrotrichlorobenzene with an aq. soln. of methylamine. I gives with concd. HNO₃ the nitroamine, 2,4,6-trinitro-1,3,5-tris(methylamino)benzene, which is a very powerful explosive, stronger than (etyl). M. Wojciechowski

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CO

Problems of modern engineering and of technology in
the light of the Chemical Engineering Congress in London.
Tadeusz Urbanski. *Przemysl Chem.* 21, 124-132 (1947).
A. C. Z.

ASSOCIATE METALLURGICAL LITERATURE CLASSIFICATION

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[illegible]

24

Photographic study of the detonation of explosives.
Detonation of trinitrotoluene. T. Urtasinski. *Roczniki*
(*Chem.* 18, 832-4 (in French, 835) (1938).—Pictures were
taken at intervals of 0.001 sec. The primary flame had a
different shape and color than the secondary one, the
latter giving more red and infrared light. M. W.

1ST AND 2ND COLUMNS																										3RD AND 4TH COLUMNS																									
PROCESSES AND PROPERTIES INDEX																																																			
<p><i>Ca</i> <i>2f</i></p> <p>Influence of ultraviolet rays on a few explosives. T. Urbanski and W. Malendowicz. <i>Pociski Chem.</i> 10, 246-253 (in French, 263-4) (1938).—The influence of ultraviolet rays on nitroglycerin, nitropentaerythritol, hexo- gen, and nitrocellulose and nitroglycerin powders was studied. The decompn. took place with const. rate. The decompn. decreased in the order nitroglycerin, nitroerythritol, nitromannitol and nitrocellulose. In general, the smaller the mol. wt. of the compd. the higher the rate of decompn. The more nitro groups there are in a mol., the more rapidly does decompn. proceed. M. W.</p>																																																			
<p>ASB-51A METALLURGICAL LITERATURE CLASSIFICATION</p>																																																			

1ST AND 2ND DEPT'S										3RD AND 4TH DEPT'S									
COMMON ELEMENTS																			
COMMON VARIABLES INDEX																			
<div style="display: flex; justify-content: space-between;"> ed 24 </div> <p>Some properties of explosive mixtures. Tadema: Urbanski. <i>Przemysl Chem.</i> 22, 621-32(1938).—A review with 80 references. Edward A. Ackermann</p>																			
<div style="display: flex; justify-content: space-between;"> OPEN COMMON ELEMENTS </div>																			
<div style="display: flex; justify-content: space-between;"> MATERIALS INDEX ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION </div>																			
1ST AND 2ND DEPT'S										3RD AND 4TH DEPT'S									
1ST AND 2ND DEPT'S										3RD AND 4TH DEPT'S									

24

The preparation and properties of nitrated starch. T. Urbanski and J. Hackel. *Congr. intern. tech. chim. ind. 4th Congr. rend., Vth congr., Budapest 2, 766-73 (1939); *Chem. Zentr.* 1940, I, 1034-5. — Earlier work (cf. C. A. Chem. Zentr. 31, 8657^a; 31, 8657^b) is summarized and new expts. are reported. Starch preps. having different viscosities gave nitrostarches having almost the same viscosities. By fractionating nitrostarch from an acetone-water mixt. 8 fractions were obtained which had somewhat different N contents but almost the same viscosities. M. G. Moore*

BC

B-11-5

Nitration of cellulose with nitric acid in presence of inorganic salts. T. URAKAWA and W. SETPOWEN (Recs. Chem., 1939, 90, 287-292). — The N content of the product of nitration of cellulose is raised from 15.0 to 15.5% by addition of 8% of H_2SO_4 , K_2HPO_4 , or $Na_2S_2O_8$, or Na_2SO_4 , but falls to very low levels as the KNO_3 or NH_4NO_3 content of system exceeds 25%. The stability of the products obtained in presence of sulphates is > when H_2SO_4 is used. R. T.

CA

10

Possible dimorphism of trinitrobenzenes. Radcliffe and J. Simon. *Rossini Chem.* 19, 457-91(1939).
 Nitration of m - $\text{C}_6\text{H}_4(\text{NO}_2)_2$ with HNO_3 and 60% oleum gave 1,3,5- $\text{C}_6\text{H}_2(\text{NO}_2)_3$ (I), m. 121°, or a substance, m. 121-2°, presumably identical with that described by Radcliffe and Pollitt (C. A. 11, 2324) as being a polymorph of I. This product is shown to be a mixt. of m - $\text{C}_6\text{H}_4(\text{NO}_2)_3$, 35-50 and 150-65%. B. C. P. A.

34

NEW DEVELOPMENTS IN THE FIELD OF RUBBER VULCANIZATION.
T. Urbanski. *Przemysl Chem.* 3, 231-3(1947)(in Polish).
The possibility of vulcanizing rubber with phenol-
formaldehyde resins is discussed on the basis of the work of
Hulfsch, v. Ruler, Cunneen, Farmer, Koch, and van der
Meer.

ASB-5LA METALLURGICAL LITERATURE CLASSIFICATION

SECTION	SUBSECTION	SECTION	SUBSECTION
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CA

PROCESSIES AND PROPERTIES. NLEP

The effect of ultraviolet light on explosives. II. Tad-
 czer Urbański (Inst. Technol., Warsaw). *Rozprawy
 Chem.* 21, 125-3 (1977). This paper corrects and ex-
 tends some of the results of previous work (cf. C.I. 33,
 1429, 1507). The photochem. decompn. of nitrogly-
 cerin can be followed by the liberation of I from KI soln.,
 and its rate can be expressed by the equation $\log(dI/dt) =$
 $\log(dI/dt) = 0.00783t + 2.5$, where w = speed of the
 reaction, I = mg. of I liberated, and t = time in min.
 For the decompn. of nitrocellulose (11.9% N) the value of
 $\log(dI/dt)$ remains approx. const. with time. Hexogen,
 on exposure to ultraviolet light, changes color from white
 to yellow, but no volatile products capable of oxidizing KI
 are produced. Nitroglycerin exposed to ultraviolet light
 continues to decomp. after the light source is removed.
 Thus, a sample of nitroglycerin on irradiation for 10 min.
 was shaken with H₂O of pH 7.00 to give an ext. of pH
 6.24; exts. made after 4, 24, 48, and 72 hrs. had pH values
 of 5.90, 5.70, 5.32, and 5.41, resp. H. H. Semant

1ST AND 2ND ORDERS										PROCESSES AND PROPERTIES INDEX									
<p>CA</p> <p>Acetylene as a raw material. T. Urbański. <i>Przemysł Chem.</i> 26, 153-64(1947).—A review of the phys. and phys.-chem. properties of C_2H_2 and of its use as raw material for the production of various materials. I. S.</p>										10									
<p>ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>										<p>1901 430899</p> <p>1911 101 044 181</p>									

118

CA

Chemistry of pyridoxine (vitamin B₆). 1. Urtanagi, *Prosymd Chem.* 20, 193-201(1947).—Early work on Vitamin B₆ is summarized. The present state of knowledge on the chemistry of pyridoxine is described. Japanese workers have heated hexoses with NH₃ salts in an autoclave at 150-160°. From the products of the reaction were derived the following 3 formulas:

CC1=CN(C=C(C=C1)O)O
(I)

CC1=CN(C=C(C=C1)O)CO
(II)

CC1=CN(C=C(C=C1)O)C
(III)

These 3 formulas are deriva. of 3-pyridol. II contains a CH₂OH group characteristic of pyridoxine. This work indicates the reaction of sugars and NH₃ salts to compds. similar to vitamin B₆ and suggests a similar reaction in living organisms. U. advances a suggestion relative to the biosynthesis of pyridoxine from formaldehyde and N compds. This suggestion is based on the work of Japanese workers on the formation of 3-pyridol deriva. indicated above and upon U's work on the condensation of 3-pyridol with formaldehyde to 6-(hydroxymethyl)-3-pyridol. The production of ψ -pyridoxine or pyridoxamine in a living organism is discussed. Since pyridoxine plays a role in Fe metabolism, its use in the treatment of anemia is also discussed.

Jeannette Skarbek

ASR-5LA METALLURGICAL LITERATURE CLASSIFICATION

GROUPS

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ca

11g

Chemotherapy of tuberculosis. T. Uriagaski. *Pres-myst Chem.* 4, 143-46(1948). - The various chemotherapeutic agents used to combat tuberculosis are described and the results obtained to date reviewed. 18 references. Frank Conet

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

CA

Recent developments in the field of explosives.
Tadeusz Urbanski. *Przemysl Chemic.* 4, 487-501(1948).--
New developments in the production of high and per-
missible explosives, detonators, and rocket fuels are re-
viewed. 33 references. Frank Conet

ASD-SLA METALLURGICAL LITERATURE CLASSIFICATION

CA

Chemotherapy of tuberculosis. H. T. Urbanski. *Pres-
myl Chem.* 5(28), 457-60(1949); cf. *C.A.* 42, 786(c).—Re-
cent advances in the chemotherapy of tuberculosis are re-
viewed and the structure and phys. properties of a large no. of
agents are described. 39 references. Frank Conet

CA

1 H

Salicylhydroxamic acid as an antitubercular agent.
 T. Urbanski (Inst. Technol., Warsaw, Poland). *Nature*
 66: 267-8(1950).—Salicylhydroxamic acid, (prepd. ac-
 cording to Janssens, *Ber.* 22, 1272(1889)) m. 168°
 (slow heating), 176-8° (quick heating), was converted to
 its monosodium salt by treatment with NaHCO₃. The
 Na salt was sol. in H₂O and a 0.1 N soln. gave pH 7.7.
 The bacteriostatic concn. of the Na salt was 1.0 mg. per
 100 ml., the same as sodium *p*-aminosalicylate, *in vitro*
 in Youmans medium against *Mycobacterium tuberculosis*
 (H₃R₂ strain). The compds. were tested *in vivo* by using
 white mice inoculated (intravenously) with 0.1 mg. of *M.*
tuberculosis. The compd., no. of animals, daily dose
 (mg. per mouse), mortality, av. tuberculous index, and
 av. survival time (days), resp., were: sodium *p*-amino-
 salicylate, 50, 10, 50, 58, and 23.1; sodium salicylhydrox-
 amate, 50, 5, 63, 68, and 22.1; control, 50, —, 100, 100,
 and 20.1. The min. lethal dose of sodium salicylhydrox-
 amate on white mice was about 12 times the curative dose.
 Wesley H. Hartung

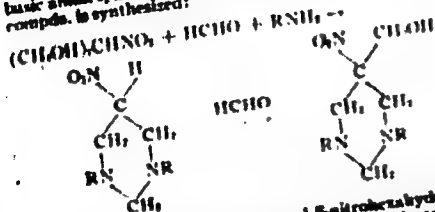
URBAŃSKI, T.

Salicylhydroxamic acid as a possible antituberculous agent. Gruzlica,
Warszawa 18 no.2:206-208 Apr-June 1950. (CML 20:7)

1. National Institute of Tuberculosis, Lodz.

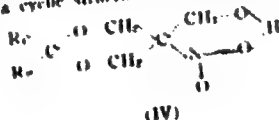
CA

Reactions of nitroarenes. IV. Reaction of nitroarenes with formaldehyde and amines. Stanislaw Malinowski and Tadeusz Urbanski (Politech. Warsaw, Poland). *Roczniki Chem.* 25, 183-212 (1951); cf. C.A. 43, 1755; 44, 4876. — The reactions between MeNO₂, HCHO, and NH₃, even in dil. soln. and 0° lead to polymers of basic character. Replacing MeNO₂ by EtNO₂ gives the same results. However, from a substituted nitromethane and less basic amines, a series of hexahydropyrimidine and α -dioxane compounds is synthesized:



When R is PhCH₂, 1,3-dibenzyl-5-nitrohexahydropyrimidine, m. 57°, and 1,3-dibenzyl-5-nitro-6-(hydroxymethyl)hexahydropyrimidine, m. 124° (HCl salt, m. 150°; diacetate, m. 162°), are formed in an exothermic reaction. When R is is-Pr, 1,3-diisopropyl-5-nitro-6-(hydroxymethyl)hexahydropyrimidine, m. 140° (HCl salt, m. 145°; diacetate, m. 159°), is isolated, and when R is cyclohexyl, 1,3-dicyclohexyl-5-nitro-6-(hydroxymethyl)hexahydropyrimidine, m. 104°, is formed. The monomers of tri-(hydroxymethyl)nitromethane (I) react slower than the

above compounds, requiring heating to 60°, and their hydrolysis during the reaction leads to the same products as from the bis(hydroxymethyl) deriva. (II). I are synthesized by conversion of (CH₂OH)₂CNO₂ (III) to acetals or ketals; these are esterified and hydrolyzed (dil. HCl/EtOH). III reacts with H₂O (steam-bath, trace of concd. H₂SO₄) to give 2-phenyl-5-(hydroxymethyl)-5-nitro- α -dioxane, m. 125-6°, and is esterified to the acetate, m. 141°, and benzoate, m. 116-17°. III with anhyd. Me₂O in the presence of anhyd. CuSO₄ at room temp. produces 2,2-dimethyl-5-(hydroxymethyl)-5-nitro- α -dioxane, m. 133-4°; acetate, m. 64°; or benzoate, m. 117-18°. PhCH₂CHO and III (steam bath, trace of concd. H₂SO₄) form (2-phenyl-5-(hydroxymethyl)-5-nitro- α -dioxane, m. 137-8°. Acetals and ketals are resistant to alk. hydrolysis (boiling with 15% KOH or NaOH) while acid hydrolysis (5% HCl at 60°) is complete in 5 min.; they are neither methylated by MeSO₃Na, nor oxidized by alk. KMnO₄ or H₂O₂; on heating with MeONa, no HCHO is released. Also no acetals are formed from II. Therefore a cyclic structure (IV) is suggested. The alk



[illegible]

URBANSKI, Tadeusz

reject

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

③ Chem.

Penta- and hexanitrate of sorbitol. Tadeusz Urbanski and Stanisława Kwiatkowska (Inst. Technol., Warsaw). *Roczniki Chem.* 25, 312-14 (1951) (English summary). The nitration of sorbitol gives a mixt. of penta- and hexanitrate; however, a nitration temp. of -10° favors the formation of the penta deriv., while the hexa deriv. is formed at $3-5^{\circ}$. To 15 g. sorbitol in 75 g. HNO_3 (d. 1.52), 150 g. H_2SO_4 (or 20% oleum) was added dropwise at $0-3^{\circ}$ for 1.5 hrs.; from the cryst. 29.7 g. product, m. $38-45^{\circ}$, was obtained 8 g. sorbitol hexanitate, m. $54.5-55^{\circ}$, by fractional crystn. from 50% aq. alc.; from the fraction easily sol. in 50% aq. alc., was pptd. 2 g. pentanitate by adding H_2O to make an approx. 35% alc. soln. Sorbitol (5 g.) added dropwise to 50 g. concd. H_2SO_4 at -10 to -70° gave 7.5 g. of a mixt., mostly pentanitate. The hexanitate is easily denitrated by warming it in alc. pyridine. Gene A. Wozny

11 M

CA

Antitubercular activity of some 8-hydroxyquinoline derivatives. T. Urbanski, S. Slopek, and J. Venulet (Inst. Technol., Warsaw, Poland). *Nature* 166, 29(1951). —T28 was prepd. by action of NaHSO_3 on 6-nitroso-8-hydroxyquinoline to yield the monohydrate of N-sulfo-N-[5-quinolyl]-8-hydroxy]hydroxylamine. *In vitro* expts. on rat heart showed T28 has no effect on the heart when 0.1 ml. of 5% soln. is administered. Intravenous doses of 50 mg./kg. body wt. to rabbit showed little effect. Guinea pigs inoculated with *Mycobacterium tuberculosis* and subsequently treated with T28 showed a tubercular index of 0.4, as compared with streptomycin 57. Other derivs. tested were 8-sulfo-8-hydroxyquinoline, 8-hydroxyquinoline sulfate, 5-amino- and 5,7-diaminoquinoline. The latter two were prepd. by reduction with NaHSO_3 of 5-nitroso- and 5,7-dinitro-8-hydroxyquinoline. T. J. Winnick

1953

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URBANSKI, T.

✓ Urbański T., Chechelska B. Preparation on Laboratory Scale of Ergosterol from Mycelium *Aspergillus Niger*.

„Otrzymywanie ergosterolu z grzybní *Aspergillus Niger* w skali laboratoryjnej”. (Prace Gl. Inst. Przem. Roln. i Spoż. No. 3) Warszawa, 1952. PWT. 4 pp., 1 tab.

The content of ergosterol in *Aspergillus Niger* taken from a citric acid factory was determined. Two principal methods of extraction of ergosterol were used: 1) extraction of fats and sterols and the hydrolysis of fats, followed by the isolation of ergosterol from the nonhydrolyzed portion, 2) hydrolysis of the whole material with potassium hydroxide in hydrated alcohol and the extraction of the unhydrolyzed portion with a suitable solvent. The evaporation of the solvent left the residue of ergosterol. In order to find the most convenient parameters, a number of experiments, in modification of both methods were carried out: the quantity of potassium hydroxide, the time of hydrolysis and extraction, the kind and the quality of solvent. The most suitable method was found to be: hydrolyzing the fungus with KOH in hydrated alcohol, using 30% of KOH — calculated on the basis of the dry fungus — mixing with water, filtering the precipitate and extracting the solution three times with benzene. Then the fungus was extracted three times with alcohol and the solution was filtered hot. The precipitate from benzene and alcoholic extract was recrystallized several times from the mixture of alcohol and benzene (4:1) or dichloroethane. Different kinds of fungi yielded various quantities of ergosterol. It is possible that the yield depends on the conditions of fermentation (medium, aeration) and of the age of fungi. The content of the crude ergosterol was 0.12 — 0.17%. After purification, it decreased to 0.08—0.12%. As the tests were executed on laboratory scale i.e. with small quantities of fungus, the losses were relatively great.

Polish Technical Abst.

No. 1 1954

Chemistry and Chemical Technology

URBANSKI, TADEUSZ

Chemical Abst.
Vol. 48 No. 3
Feb. 10, 1954
Organic Chemistry

Phenolic reactions of 3-hydroxypyridine. Halina Bojarska-Dahlig and Tadeusz Urbanski. *Prace Chem. Pol.* 1952, No. 1, 1-15 (English summary).—The authors undertook a systematic study of 3-hydroxypyridine (I), the prototype of vitamin B₆. The NH₄ salt of I was obtained in 75.4% yield by adding dropwise 3 moles pyridine (b. 114-16°) to 155 cc. 0.5% concn in presence of 6.5 g. Hg; the reactor, equipped with a condenser, was cooled with water, and then slowly brought to 250°, and kept at this temp. for 8 hrs. The reaction mixt. dissolved in water, neutralized, and concd. gave 308 g. NH₄ salt of I, crystals from water, m. 238-42°. The NH₄ salt of I, crystals from water, m. 238-42°. The NH₄ salt (1 mole) fused with 9 moles KOH for 3 hrs. at 180° gave, after neutralization and extn. with Me₂CO, 80% of I, m. 124.5-5.6°, b₁₀ 207.5-9.0°; picrate, m. 200-1.5° (from alc.); picrolonate, C₁₁H₈ON.C₆H₄O₂N₂, m. 241.5-3.0° (from alc.); C₁₁H₈ON.HgCl₂, m. 162.0-2.5° (from water); C₁₁H₈ON.Cu(OAc)₂, m. 180-3° (from water). Carboxylation of I gave hydroxypicolinic acids. Dry Na salt of I and CO₂ heated rapidly to 250° at atm. pressure gave 8-9.6% 3-hydroxypicolinic acid (II). With slow heating less or no II was obtained. At 215-20° and 45 atm. the yield of II was 22%. Same conditions with K salt of I gave 24% 3-hydroxypicolinic acid (III) and 3% of II. I mixed with anhyd. K₂CO₃ treated with CO₂ at 215° and 45 atm. for 8-9 hrs. gave III in yields which depended on the ratio of K₂CO₃ to 3-hydroxypyridine: ratio 1.5 gave highest yield, 85-7%; ratio 1.0 gave 70%. HgCl₂ salt of II, crystals from water, m. above 220° (decolor.). II picrate m. 158.5-62.0° (from water). Me 3-hydroxypicolinate, m. 73-4°. Et 3-hydroxypicolinate (IV), b₁₀ 124°, b₁₀ 152-4°, b₁₀ 162°; HgCl₂ salt, m. 147-7.5° (from water), picrate, m. 118-19.5° (from water). HgCl₂ salt of III, m. 254-4° (from water). III picrate m. 205-0.5° (from water). Me 5-hydroxypicolinate m. 71.5-3.0° (from CHCl₃); HgCl₂ salt, m. 163.5-4.5° (from water). Esters and ethers of I were prepd. and characterized. I and Ac₂O gave 95% 3-pyridyl acetate, b₁₀ 92°, b₁₀ 137°; picrate, m. 155.5-7.0° (from alc.); picrolonate, m. 166.5-7.5° (from alc.). HgCl₂ salt, m. 148-0° (from water). I and BaCl gave 81% 3-pyridyl benzoate, m. 50-50.5° (from alc.); picrate, m. 152-3° (from CHCl₃); HgCl₂

salt, m. 168-70°. I and Me_2SO , gave 92% 3-pyridyl Me ether, b. 178-9°; HgCl_2 salt, m. 110-11°. K salt of I and PhBr in presence of I and CuClO_4 gave 3-pyridyl Ph ether (V). Best yields (55-60%) of V were obtained with 1-PhBr molar ratio of 1:1.5, 25% excess of I over its K salt, 2.6 g. CuClO_4 per mole of K salt, and heating reaction mixt. for 3 hrs. at 150° and 15 hrs. at 180°. V hydrochloride m. 95-7°, picrate, m. 130-2° (from alc.), picronolate, m. 102-4° (from alc.); HgCl_2 salt, m. 70-75° (from alc.). I coupled with $p\text{-O}_2\text{NC}_6\text{H}_4\text{NCl}$ and the azo compd. reduced with SnCl_4 gave 54.5% 2-amino-3-hydroxypyridine, m. 163-7.5° (from $\text{C}_6\text{H}_5\text{alc.}$); picrate, m. 257°. Iodination of I in H_2SO_4 alk. soln. gave 90% 2-iodo-3-hydroxypyridine, m. 105-6° (from MeOH). I failed to undergo nitrosation, reaction with $\text{CH}_3\text{ClCO}_2\text{H}$ to give (3-pyridoxy)acetic acid, or the Reimer-Tiemann reaction. $\text{C}_6\text{H}_5\text{N.Hg}(\text{NO}_3)_2$ did not give hydroxynitropyridine with HNO_3 .
Janina R. Spencer

URBANSKI, T; SERAFINOWA, B; MALINOWSKI, S; SLOPEK, S; KAMIENSKA, I; VENULET, J;
JAKIMOWSKA, K.

Research on new drugs for the treatment of tuberculosis. *Gruslica*,
Warsz. 20 no.2:157-170; cont'd. Mar-Apr 1952. (GLML 22:3)

1. Of the Department of Chemotherapy of the Institute of Tuberculosis (Director--Prof. J. Misiewicz, M. D.).

URBANSKI, T.; SERAFINOWA, B.; MALINOWSKI, S.; SLOPEK, S. KAMINSKA, I.; VENULET, J.;
JAKIMOWSKA, K.

Research on new drugs in the treatment of tuberculosis; thiosemicarba-
zones. Gruslica, Warsz. 20 no.3:292-302; concl. May-June 1952.
(CIAM 23:2)

1. Of the Chemical Laboratory of the Institute of Tuberculosis (Director
--Prof. J. Misiewicz, M.D.), Warsaw.

URBANSKI, T.; MALINOWSKI, S.

Synthesis of antituberculous drugs at the Institute of Tuberculosis.
Gruslica 20:6 Suppl. 2:81-91 1952. (CIAML 24:2)

1. Of the Laboratory of the Syntheses of Therapeutics of the Institute
of Tuberculosis, Warsaw.

CRBAŃSKI, TADEUSZ

Chemistry of pyridine. III. On the carboxylation of 3-hydroxypyridine with carbon dioxide. Halina Bojarska-Hablik and Tadeusz Urbaniak (Inst. Technol., Warsaw). *Roczniki Chem.* 26, 168-69 (1952) (English summary); cf. C.A. 41, 6139d; 49, 1033a. —NH₂ 3-pyridinesulfonate (I mole) was fused with KOH (2 moles) at 180° for 3 hrs., the melt dissolved in water, adjusted to a pH of about 10 with concd. HCl and then to about 4.5 with HOAc, and the resulting ppt. extrd. with Me₂CO to give 80% 3-hydroxypyridine (I), m. 124.5-5.0° (from water); *HCl* salt, b₁ 201-6° and b₂ 207-0°; *picrate*, m. 206.0-1.5°; *picrolonate*, m. 241.5-3.0°; *HgCl₂* complex, m. 162°; *Cu(OAc)₂* complex, m. 100-0° (decompn.). After I as the Na salt (II) (4.75 g.) was heated to 220°, dry CO₂ (at atm. pressure) was introduced while the temp. was raised to 250° during 30 min.; kept there 6-9 hrs., the mixt. dissolved in 25 ml. water, acidified with concd. HCl, filtered, neutralized with NaHCO₃, the unreacted I filtered off, the filtrate acidified with HOAc, and 3-hydroxypicolinic acid (III) pptd. as the Cu salt (IV) from the boiling filtrate on satn. with NaOAc. IV was decompd. with H₂S and two purifications were effected through IV to yield 0.56-0.60 g. III, m. 203-4°; *picrate*, m. 159-62°; *HgCl₂* complex, m. 220° (decompn.). III (2.6 g.) as the Ag salt was refluxed 3.5 hrs. with 1.25 g. EtI in 21 ml. dry C₆H₆, the AgI filtered off, and the benzene soln. evaporated to yield 2.39 g. Et 3-hydroxypicolinate, b₁ 162° and b₂ 124°; *picrate*, m. 118-19°; *HgCl₂* complex, m. 147°. Dry CO₂ was introduced to 1.9 g. I, as the Na salt, in a 50-ml. autoclave to a pressure of 40-45 atm., the whole heated during 1 hr. to 210-20°, kept at this temp. for 9 hrs., and the resulting mixt. worked up as

above yielded 0.62 g. III. I (1.9 g.) as the K salt was treated with CO₂ under pressure, the mixt. heated as above 8 hrs., the product dissolved in 20 ml. water, the soln. sepd. from carbonized substances by filtration, neutralized with concd. HCl, and acidified with HOAc to give crystals of 3-hydroxypicolinic acid (V). The filtrate was neutralized with NaHCO₃, any sepd. I filtered off, and after acidifying with HOAc, the Cu salts of the 3-hydroxypicolinic acids were pptd. with Cu(OAc)₂ from hot soln. The Cu salts were decompd. with H₂S and two fractions with different water solubilities were obtained. The less sol. fraction consisted of V (0.75 g.); the total yield of V was 24%, m. 267-8°; *picrate*, m. 265.0-6.6°; *HgCl₂* complex, m. 253-4°; *Me ester*, m. 72-3°; *HgCl₂* complex of *Me ester*, m. 194°. The more sol. fraction was III (3%). I (0.02 mole) was mixed with K₂CO₃ (0.03 mole) and the mixt. treated in an autoclave with dry CO₂ (40 atm.) at 215° for 8.5 hrs. From the reaction product, worked up in the usual way, was obtained 2.73 g. V. When I and K₂CO₃ were mixed in molar amts. and treated as above 70% V and 1% III were obtained. Larger excesses of K₂CO₃ did not improve the yield of V.

T. T. Galkowski

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Urbanski, Tadeusz

112

Reactions of nitroparaffins. VII. Reactions of nitro-
methane, with acetaldehyde and amines. Zygant-
stem and Tadeusz Urbanski (Inst. Technol., Warsaw,
Poland). *Russk. Khim. Pr.* 1931, 10, 152 (English sum-
mary); cf. C 1 46, 7931c. Addn. of AcH to MeNO₂ gave
either MeCH(OH)CH₂NO₂ (I) or O₂NCH₂CH₂Me (II),
depending on the reaction conditions. AcH (88 g.) in 80 ml.
water added with cooling to 15°. MeNO₂ and 0.1 g. K₂CO₃
(more K₂CO₃ was added during the reaction to keep the pH
at 7.5-8.0) and the mixt. heated 3-6 hrs. at 40° gave 75-
80% I, extd. with 1 l. Et₂O and purified through the Na
salt, m. 70-74°/0.2-0.3 mm., *n*_D²⁰ 1.412. Chlori-
nation of the Na salt of I gave 72-81% crude MeCH(OH)CH-
ClNO₂ (III), b.p. 89-91°. The Na salt (187 g.) of I in 800
ml. CCl₄ and 76.5 ml. Br in 200 ml. CCl₄ kept below 0° gave
76.5-81.5% crude MeCH(OH)CHBrNO₂ (IV), m.p. 1-10°, I
and IV cause skin burns and severe eczema. AcH (100 g.) in
236 ml. water added to 244 g. MeNO₂ and 1 g. C₂H₅OH with
cooling to 30-35°, and the mixt. stirred 2-3 hrs., then left at
room temp. 48 hrs. in a tightly closed vessel (pH = 6.5-7.0
at end of the reaction), treated with CO₂ and 6-7 l. Et₂O, and

the dried Et₂O layer distil. at 0.1 mm. Hg pressure gave 50-
95% (based on MeNO₂) crude II, not crystd., *n*_D²⁰ 1.4695.
AcCl (25 g.) and 15 g. II in 70 ml. CHCl₃ reduced until
evolution of HCl ceased gave, after distn., a residue of semi-
cryst. product which, filtered, yielded 8.6 g. O₂NCH₂(CH-
Me)₂ (V), m.p. 87-88° (from alc.) III and AcH gave
61.3% O₂NCH₂(CHMe)₂ (V), m. 118-20°. Freely
distd. AcH (18 ml.) in 18 ml. water and 36.8 g. IV neutral-
ized (litmus) with aq. KOH, the mixt. stood overnight,
salted out with NaCl, extd. with Et₂O, and the product
crystd. from CCl₄ and CHCl₃ (1:1) gave white needles
of O₂NCH₂(CHMe)₂, m. 111-113°. Cyclic acetals (in or-
ranes) of the type O(CHMe.CRR'₂NO₂)₂CHMe.O.CRR'₂

(VA) were prepd. from II by 3 methods. (1) 1.4 mole crude
II and 1.5 moles aldehyde was added to 2 g. concd. H₂-
SO₄, the mixt. heated several hrs. at 60-80°, and the product
isolated by addn. of Et₂O or alc.; (2) 1 mole crude II or V
and 1.5 moles aldehyde in C₂H₅ were added 0.65 g. g.
PhSO₃H, alc. HCl, and H₂SO₄, the water was decanted
throughout the course of the reaction, and the product
isolated as in (1). The following 2-substituted-2,2'-bis-
methyl-*o*-dioxanes (VA, R¹ = R² = H; R¹, m.p., and 1%
yield given) were prepd. by method (1): Me (VI), 65-67°,
71.4; CH₃ (VII), 101-102°, 25.7; Ph (VIII), 120-121°, 12.5
[37.2% by method (2)]; p-O₂NCH₂ (IX), 116-117°, 11.0;

299 MUNT ECKSTEIN

7

212

p -HOC₆H₄ (X), 222-4°, 15.7; p -MeOC₆H₄ (XI), 132-134°, 14.9; p -HCH₂CH₂ (XII), 84.5-86°, 3.7; p -Me₂NC₆H₄ (XIII), 181-3°, 8.9. In the prepn. of XI-XIII unreacted aldehyde was removed with aq. NaHSO₃ before isolation of the product. The VA were crystd. from alc. (VII, IX, XII, XIII), from aq. (1:1, 1:3) alc. (VI, XI), or from a mixt. (3:2) of C₆H₆ and alc. (X). They are resistant to dil. alkali (except VII), but hydrolyze in dil. acids. They give pseudonitroles with RNO₂, while II with HNO₃ splits off AcH to give the nitrolic acid. The following VA (R¹ = R² = H; R¹, m.p., and % yield given) were prepd. by method (2): p -ClC₆H₄, 107-9°, 36.4; o -HOC₆H₄ (XIV), 144-6°, 21.2; p -AcNHC₆H₄, 206-7.5°, 13.2. *m*-Dioxanes (VA, R¹ = H; R², m.p., and % yield given): p -O₂NC₆H₄, Br, 142-4°, 32.5; p -MeOC₆H₄, Br, 124-6°, 9.2; Ph, Br, 98-100°, 36.5; p -O₂NC₆H₄, Cl, 125-7°, 55.6; Ph, Cl, 91-2.5°, 36.5. All the VA derived from 4,6-dimethyl-*m*-dioxane are white cryst. compds. except XIII, which is yellow. XIV required 16 hrs. for its prepn. and could be made by method (2) only. VA [R¹ = R² = Me, R³ = H (XV), and R¹ = Me, R² = Pr, R³ = H (XVI)] were prepd. Crude II (150 g.), 600 ml. dry Me₂CO, 200 g. anhyd. CuSO₄, and 1 ml. alc. HCl (30%) refluxed 24 hrs., the sulfate filtered off, and the Me₂CO distd. gave an oily residue which, washed with water and distd. up to 70°/5 mm., yielded a cryst. residue of 40 g. (21.1%) XV, needles with camphoric odor, m. 73.5-5.5° (from alc.). II (30 g.), 80 g. PrAc, 100 g. anhyd. CuSO₄, and 1 drop alc. HCl 16 hrs. at 100° gave 15 g. (28%) XVI, needles with camphoric odor m. 68-70° (from 1:1 aq. alc.). Attempts to synthesize tetrahydrooxazine or hexahydropyrimidine derivs. from II and primary amines were unsuccessful since II decomd., splitting off AcH and forming resinous polymers. Cyclohexylamine and III gave AcH and a cryst. unstable product, m. 62-4°, presumably the salt of cyclohexylamine and the hydroxamic acid [C(C(O)NHOH)].

Ludwig R. Spruener

Reactions of aromatic azides with azanoguanidine. Formation of aryl derivatives of amidinoazides and their transformation into carbonates. T. I. Gerasim, B. N. Krasnaya, N. A. Zhukova, H. H. Hukhlova, and V. I. Zhukovskii, *Izv. Akad. Nauk SSSR Khim. Tekhnol. Khim. Pril.*, 1967, 1066, 1067, 1068, 1069, 1070, 1071, 1072, 1073, 1074, 1075, 1076, 1077, 1078, 1079, 1080, 1081, 1082, 1083, 1084, 1085, 1086, 1087, 1088, 1089, 1090, 1091, 1092, 1093, 1094, 1095, 1096, 1097, 1098, 1099, 1100, 1101, 1102, 1103, 1104, 1105, 1106, 1107, 1108, 1109, 1110, 1111, 1112, 1113, 1114, 1115, 1116, 1117, 1118, 1119, 1120, 1121, 1122, 1123, 1124, 1125, 1126, 1127, 1128, 1129, 1130, 1131, 1132, 1133, 1134, 1135, 1136, 1137, 1138, 1139, 1140, 1141, 1142, 1143, 1144, 1145, 1146, 1147, 1148, 1149, 1150, 1151, 1152, 1153, 1154, 1155, 1156, 1157, 1158, 1159, 1160, 1161, 1162, 1163, 1164, 1165, 1166, 1167, 1168, 1169, 1170, 1171, 1172, 1173, 1174, 1175, 1176, 1177, 1178, 1179, 1180, 1181, 1182, 1183, 1184, 1185, 1186, 1187, 1188, 1189, 1190, 1191, 1192, 1193, 1194, 1195, 1196, 1197, 1198, 1199, 1200, 1201, 1202, 1203, 1204, 1205, 1206, 1207, 1208, 1209, 1210, 1211, 1212, 1213, 1214, 1215, 1216, 1217, 1218, 1219, 1220, 1221, 1222, 1223, 1224, 1225, 1226, 1227, 1228, 1229, 1230, 1231, 1232, 1233, 1234, 1235, 1236, 1237, 1238, 1239, 1240, 1241, 1242, 1243, 1244, 1245, 1246, 1247, 1248, 1249, 1250, 1251, 1252, 1253, 1254, 1255, 1256, 1257, 1258, 1259, 1260, 1261, 1262, 1263, 1264, 1265, 1266, 1267, 1268, 1269, 1270, 1271, 1272, 1273, 1274, 1275, 1276, 1277, 1278, 1279, 1280, 1281, 1282, 1283, 1284, 1285, 1286, 1287, 1288, 1289, 1290, 1291, 1292, 1293, 1294, 1295, 1296, 1297, 1298, 1299, 1300, 1301, 1302, 1303, 1304, 1305, 1306, 1307, 1308, 1309, 1310, 1311, 1312, 1313, 1314, 1315, 1316, 1317, 1318, 1319, 1320, 1321, 1322, 1323, 1324, 1325, 1326, 1327, 1328, 1329, 1330, 1331, 1332, 1333, 1334, 1335, 1336, 1337, 1338, 1339, 1340, 1341, 1342, 1343, 1344, 1345, 1346, 1347, 1348, 1349, 1350, 1351, 1352, 1353, 1354, 1355, 1356, 1357, 1358, 1359, 1360, 1361, 1362, 1363, 1364, 1365, 1366, 1367, 1368, 1369, 1370, 1371, 1372, 1373, 1374, 1375, 1376, 1377, 1378, 1379, 1380, 1381, 1382, 1383, 1384, 1385, 1386, 1387, 1388, 1389, 1390, 1391, 1392, 1393, 1394, 1395, 1396, 1397, 1398, 1399, 1400, 1401, 1402, 1403, 1404, 1405, 1406, 1407, 1408, 1409, 1410, 1411, 1412, 1413, 1414, 1415, 1416, 1417, 1418, 1419, 1420, 1421, 1422, 1423, 1424, 1425, 1426, 1427, 1428, 1429, 1430, 1431, 1432, 1433, 1434, 1435, 1436, 1437, 1438, 1439, 1440, 1441, 1442, 1443, 1444, 1445, 1446, 1447, 1448, 1449, 1450, 1451, 1452, 1453, 1454, 1455, 1456, 1457, 1458, 1459, 1460, 1461, 1462, 1463, 1464, 1465, 1466, 1467, 1468, 1469, 1470, 1471, 1472, 1473, 1474, 1475, 1476, 1477, 1478, 1479, 1480, 1481, 1482, 1483, 1484, 1485, 1486, 1487, 1488, 1489, 1490, 1491, 1492, 1493, 1494, 1495, 1496, 1497, 1498, 1499, 1500, 1501, 1502, 1503, 1504, 1505, 1506, 1507, 1508, 1509, 1510, 1511, 1512, 1513, 1514, 1515, 1516, 1517, 1518, 1519, 1520, 1521, 1522, 1523, 1524, 1525, 1526, 1527, 1528, 1529, 1530, 1531, 1532, 1533, 1534, 1535, 1536, 1537, 1538, 1539, 1540, 1541, 1542, 1543, 1544, 1545, 1546, 1547, 1548, 1549, 1550, 1551, 1552, 1553, 1554, 1555, 1556, 1557, 1558, 1559, 1560, 1561, 1562, 1563, 1564, 1565, 1566, 1567, 1568, 1569, 1570, 1571, 1572, 1573, 1574, 1575, 1576, 1577, 1578, 1579, 1580, 1581, 1582, 1583, 1584, 1585, 1586, 1587, 1588, 1589, 1590, 1591, 1592, 1593, 1594, 1595, 1596, 1597, 1598, 1599, 1600, 1601, 1602, 1603, 1604, 1605, 1606, 1607, 1608, 1609, 1610, 1611, 1612, 1613, 1614, 1615, 1616, 1617, 1618, 1619, 1620, 1621, 1622, 1623, 1624, 1625, 1626, 1627, 1628, 1629, 1630, 1631, 1632, 1633, 1634, 1635, 1636, 1637, 1638, 1639, 1640, 1641, 1642, 1643, 1644, 1645, 1646, 1647, 1648, 1649, 1650, 1651, 1652, 1653, 1654, 1655, 1656, 1657, 1658, 1659, 1660, 1661, 1662, 1663, 1664, 1665, 1666, 1667, 1668, 1669, 1670, 1671, 1672, 1673, 1674, 1675, 1676, 1677, 1678, 1679, 1680, 1681, 1682, 1683, 1684, 1685, 1686, 1687, 1688, 1689, 1690, 1691, 1692, 1693, 1694, 1695, 1696, 1697, 1698, 1699, 1700, 1701, 1702, 1703, 1704, 1705, 1706, 1707, 1708, 1709, 1710, 1711, 1712, 1713, 1714, 1715, 1716, 1717, 1718, 1719, 1720, 1721, 1722, 1723, 1724, 1725, 1726, 1727, 1728, 1729,

6. *Chilomeniscus* S. Kne. 1833

1. 1. The first

URBANSKI, T.

"On the Structure of Some Aliphatic Nitro Compounds." In English. P. 239,
(GEODEZJA I KARTOGRAFIA, Vol. 1, No. 6, 1953, Warszawa, Poland.)
(Polska Akademia Nauk.)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 3,
No. 12, Dec. 1954, Uncl.

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Hydrazine Acids III Assays of salicylhydrazinic
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following new hydrazine acids all possess anti-inflammatory and bacteriostatic action on mycobacteria, particularly complete on T106 and T139. *Salicylhydrazinic acids*

T-85	T-85	m 119-50°	4-Hz (T-97)	m 100-2°	5-Hz
T-95	45°	1-Hz m 112	m 154-6°	1-Hz	T143
T-96	44°	1-Hz m 112	m 154-6°	1-Hz	T143

The above are new hydrazine acids.

Products T-85, T-97, T100, T112, and T143 were also checked according to Jean Renaud [Rev. 23, 1270-1289]. Yields for the micilyc derive ranged from 35 to 90%. Yields averaged 25% for T106 and T139. Products T151, T106, and T139 were synthesized from NH₄OH in alc. KOH with the Me esters of the corresponding CO₂H acids.

Clayton F. Holoway

ORBAŃSKI, TADEUSZ

Reactions of cyanoguanidine with aromatic acids. 111.
 2,4-Dihydroxy-7-nitroquinazoline from 4-nitroanthranilic
 acid and cyanoguanidine. Tadeusz Orbański, Lech
 Skowrońska-Serapina and Józef Głowacki (Inst.
 Technol. Politechniki Warszaw.). *Kochemia Chem.* 27, 167-68
 (1953) (English summary); *Ch. C.I.* 48, 13017d. When
 heated with HCl, 4-nitroanthranilic acid and cyanoguanidine
 yield 2,4-dihydroxy-7-nitroquinazoline (I), tautomeric with
 2,4-dioxo-7-nitrotetrahydroquinazoline. I has been prepd.
 in another way by Hantres and Gledling (*C.I.* 37, 6509).
 Clayton F. Holoway

NO

LEWENSTEIN, JADU 27

BULG !

Investigation of hydroxamic acids. II. 5-Bromo-salicylhydroxamic acid. Jadwiga Ochalska and Wieslaw Lewenstein (Polytech., Warsaw). *Roczniki Chem.* 27, 313 (1951); cf. *C.A.* 45, 23084. D: (10 g.) in 50 ml. of glacial HOAc was added slowly to .5 g. of salicylhydroxamic acid in 100 ml. of glacial HOAc at 60°. Excess Br was removed with NaHSO₃ and, after cooling, the product was distd., washed with cold water, and dried to yield 35 g. crude 5-bromosalicylhydroxamic acid, white crystals, (from alc.), m. 232° (decompn.), poorly sol. in water and solvents such as ether, acetone, CCl₄, benzene, and xylene at boiling temps., readily sol. in boiling alc. and HOAc; it has a bitter taste. C. F. Holoway

115 244

Some derivatives of 4,4'-dihydroxydiphenyl sulfone.
 Karamer, Okon and Tadjer, *Chimica*, 1962, 1, 1, 1.
 27, 318-64 (1963) English summary. — p-Cresol (37.1 g.) is added slowly to 100 ml. of 5% H₂SO₄ at 35-40°, the mixture heated slowly to 100° for 5 hrs., the pressure reduced from 250 to 150 mm. while the temp. is raised to 180° over 30 hrs. The H₂O formed in the reaction is removed by distn. The temp. is then lowered to 110°, 2N NaOH added to pH 9, filtered, and the filtrate adjusted to pH 3.5 with HCl, resulting in 23.05 g. of a mixt. of 4,4'-dihydroxy-2,2'-bis(4-phenyl) diphenyl sulfone (I) and its 3,3'-diarylether analog, m. 202-8°. The mixt. is dissolved in boiling MeCO, twice its vol. of warm CaH₂ is added, and the mixt. is cooled, pptg. 72% I, m. 271-1.5°; concn. of filtrate yields 24% II, m. 224-1°. Similarly, pyrocatechol at 70-80° with a digestion temp. of 210° for 24 hrs. gives 28% of 3,3'-4,4'-bis(4-phenyl) diphenyl sulfone, m. 237-40°, 3,3'-Dihydroxydiphenyl sulfone (III) (50 g.) is added to 500 ml. H₂SO₄ at 20°, cooled to 15°, and 250 ml. HNO₃ is added, not permitting the temp. to rise above 35-40°. The mixt. is heated at 95-100° for 30 min., diltd. with an equal vol. of H₂O, and filtered, giving the 3,3'-diarylether analog of I, m. 251-1° (from EtOH), 31% of which is dissolved in 820 ml. of 25% aq. NH₃ at 40°, 157 g. Na₂SO₃ is added, the mixt. heated on a H₂O bath for 6 hrs. and cooled, to give 23.9 g. of the 3,3'-diarylether analog, m. 271-1°. 4,4'-Dihydroxy-2-aryldiphenyl sulfone, m. 200-201°, is obtained in 12% yield by treating salicylic acid with 250 ml. AgO with 0.1 g. NaOAc on a H₂O bath for 8 hrs. Heating 25 g. III and 250 ml. AgO with 0.1 g. NaOAc on a H₂O bath for 8 hrs. gives the diarylether analog of I, m. 170-1.5°, 14% (25 g.) is dissolved in 100 ml. 2N NaOH and 20 g. Me₂SO is added slowly, pptg. 92% 4,4'-dimethoxydiphenyl sulfone, m. 122-3° (from EtOH). III (25 g.) is dissolved in a soln. of 17 g. NaOH in 75 ml. H₂O at 50-60°, 10 g. ClCH₂COH is added, and the mixt. heated at 100° for 1 hr. The resulting ppt. is dissolved in boiling H₂O, and cooled, pptg. 3,3'-diarylether analog of I, m. 251-1°. V with 250 ml. MeOH is heated, m. 122-3°.

Hobanski Tadysz

oxyquinone and

with a

chemistry of pyridine. IV. Reactions of amides of nicotinic, isonicotinic, and isonicotinic acids with formic acid and hydrazine. *Researches in Chemistry and Technology* (Inst. Technol. Charkov) *Russkii Khim. Zh.* 27, 103 (1931) (English summary).—(1) Through at this alab. in pyridine, R' = picolinoyl, and R' = isonicotinoyl, the amide (21.4 g.) in 60 ml. H₂O, 2.2 g. K₂CO₃, and 2.0 g. 37% CH₃O are heated on a H₂O bath for 1 hr., the product is extracted with EtOH, the EtOH is distilled, and the residue is dried in a vacuum oven at 40° for 2 hrs., yielding 2.4 g. of product. Similarly 1.5 g. I treated with H₂SO₄ (3.5 g.) and 2.0 g. 37% CH₃O and 25 ml. 85% H₂SO₄ is heated at 40° for 2 hrs., poured into ice water, and Na₂CO₃ added to pH 5, the pptd. starting material is filtered off, and solid Na₂CO₃ ppts. 1.3 g. II. Also prepd.: II picrate, m. 254-5°, and III-HCl, m. 167-6°. In the same manner, R'NHCH₃CH (III), m. 124-6°, (HCl salt, m. 202-3°) and (R'NHCH₃CH₂ (IV), m. 124-6°) are obtained. R'NHCH₃CH₂Me₂HCl (V), m. 159-6°, is prepd. (a) by refluxing 2 g. R'NH₂ with 2.1 g. Me₂NH in EtOH and 2.0 g. 37% CH₃O in 16 ml. aq. EtOH for 3 hrs.; HCl, m. 160-2°. III (1.7 g.) and 1.5 g. 2-naphthalene V picrate m. 160-2°. III (1.7 g.) and 1.5 g. 2-naphthalene V picrate in 29 ml. EtOH, contg. 1 ml. conc. HCl, for 4/5 reduced in 29 ml. EtOH, the residue allowed to stand hr., the EtOH is distd., and the residue allowed to stand hr., the EtOH is distd., and the residue allowed to stand hr. for several months, giving the 2-GH₃OCH₃CH₂ (VI), m. 170-7°. By the methods described, R'NHCH₃CH (VII), m. 145-7°. (R'NHCH₃CH₂ (VII), m. 376-8°. VI picrate, m. 145-7°. (R'NHCH₃CH₂ (VII), m. 253-7°. R'NHCH₃CH₂ (VIII), m. 165-9°, and VIII picrate, m. 123-9°. VMe₂HCl (VIII), m. 165-9°, and VIII picrate, m. 123-9°. are prepd. V and VIII show a slight but noticeable action in ether against Me₂CO, but not Me₂CO and Me₂CO in ether. *Chem. Abstr.* 270.

94

Urban: Tadmor

[illegible]

URBANSKI, T.

✓ The structure of some aliphatic nitro compounds. T. Urbanski, Bull. acad. polon. sci., Cl. III, 7, 593-4 (1951) (in part); cf. C.A. 49, 8002a. — Certain aliphatic nitro compounds do not show the 270 mμ absorption characteristic of the nitro group. An explanation involving H bonding is suggested (cf. Allrecht and Corey, C.A. 33, 4845; Shugam, C.A. 45, 4112b). Two examples cited are $(Me_2C(NO_2)CH_2)_2NH^+Cl^-$ and $(MeC(CH_2OH)(NO_2)CH_2)_2NH$. J. E. A.

URBANSKI, T.

3852

547.551.43:547.495.9

Urbanski T., Skowrońska-Serafinowa B., Dąbrowska H. Reactions of
Cyanguanidine with Aromatic Amines. Preparation of New Derivatives
of Amidine-Phenyl-Urea, their Transformation into Diphenyl Urea
Derivatives.

CH

„Reakcje cyanoguanidyny z aminami aromatycznymi. Otrzymywanie
nowych pochodnych amidyno-fenilo-mocznika i przekształcenie ich
w pochodne dwufenylomocznika”, Roczniki Chemii (PAN). No. 3, 1954,
pp. 423—437.

Continuing the work on the reactions of aromatic amines with
cyanguanidine in the presence of hydrochloric acid, a number of new
derivatives of amidine-phenyl-urea were obtained, corresponding to the
general formula: $X.C_6H_4.NH.CO.NH.C(NH).NH_2$. It was found that
boiling the amidine-urea derivatives in aniline led to the splitting of
these compounds and the formation of various diphenyl-urea derivatives.
A hypothesis is advanced for a chain reaction mechanism.

11
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Urbanowski, T.

V Reactions of aromatic amides with cyanoguanidine. Formation of derivatives of *gaidinonora* and their reaction with aniline. T. Urbanowski, B. Skowronski-Skrabinczyk, and H. Dabrowska. *Pol. arch. chem. sci.*, Classe III, 2, 433-4 (1961); *J. C.A.* 49, 809. The following 3,4-XYC(1)-NHCONHC(:NH)NH₂ were prepd. (X, Y, and m.p. given): Cl, H, 143-4° (I); Br, H, 172-3° (II); NH₂, H, (HCl salt, m. 300°) (III); SO₂H, H, 267-9° (IV); SO₂NH₂, H, 312-13° (V); H, OH, → (sulfate, m. 230-2°) (VI). The compds. were made by boiling the corresponding amines with cyanoguanidine in the presence of HCl. V was formed from sulfanilamide-HCl in aq. medium without added HCl. The reaction with aniline is exemplified by: *p*-XC₆H₄NHCONHC(:NH)NH₂ + PhNH₂ → *p*-XC₆H₄NHCONHPh (VII) + (H₂N)C(:NH)NH₂; VII + PhNH₂ → (PhNH)₂CO + *p*-XC₆H₄NH₂. I-VI were tested against *Mycobacteria* for

bacteriostatic action with inhibiting concns. varying from 1-125 mg.-%, depending upon the deriv. used and the strain of organism. Howard Nechemkin

URBAN, T. H.

V
C1
In the production of adipic acid from Furfural, the following procedure is described: Furfural is treated with 10% NaOH solution (No. 2, 1942) (1954). — Furfural is gathered in July and August (see in adipic acid content of 12% dry basis). Of a no. of extraction and purification methods investigated, the following was found to be the most satisfactory: Sawdust is washed with 0.5% HCl, extd. 3 times with 2% Na₂CO₃, the ext. is bleached with ClO₂ or NaClO₂, adipic acid is pptd. with HCl and washed with water and alcohol. — Alina S. Szegedy.

15/91
C

URBANSKI, Tadeusz; MALINOWSKI, Stanislaw; SKOWRONSKA-SERAFINOWA, Barbara;
CHRECHNISKI, Bozena; DABROWSKA, Halina; PALECKI, Jerzy; GURNE,
Daniela; HALSKI, Leszek; SLOPEK, Stefan; KAMINSKA, Irena;
VENULET, Jan; JAKIMOWSKA, Krystyna; URBANSKA, Alicja

Search for new antituberculous agents. Gruslica 22 no.10:681-690
Oct 54.

1. Z Oddzialu Syntezy Lekow Instytutu Gruslicy; kierownik prof. dr.
T.Urbanski, dyrektor: prof. dr. J.Misiewica.

(CHEMOTHERAPY, in various diseases
tuberc., progr.)

(TUBERCULOSIS, therapy
antituberc. agents, research)

URBANSKI, T.

POL

Reactions of aliphatic nitro compounds. X. Formation of the hexahydroindole ring using 1-nitropropane, formaldehyde, and ammonia. Tadeusz Urbancki, Zbigniew Jurecki, and Ewa Lipiak. *Repts. Chem.* 73: 109 (1954) (English summary). 1. preceding article. Pd/C, CH_3O , and NH_3 in a 1:3:3 molar ratio at 50°C for 1.5 hrs give 3% 5-nitro-2-ethylhexahydroindole (I), m. 159-60°. A 12.5% yield of I is obtained by dissolving 5-nitro-2-ethyl-1,3-propanediol in 120 ml 25% NH_3 soln with C_6H_6 for several weeks and treating with aq. HCl to obtain I·HCl, m. 159°, which reacts with 20% NaOH to give the 1,1-dinitro deriv., m. 116°. Before this with HCl gives the dihydrochloride of I, m. 157-8°. XI. A new derivative of tetrahydroxaline with nitromethane, formaldehyde, and benzylamine. Tadeusz Urbancki and Ewa Lipiak. *Ibid.* 175-94. One mole of $(\text{HOCH}_2)_2\text{CNC}_6\text{H}_5$ and 1 mole of benzylamine with app. 3 moles of 20% CH_3O at 15° stirred for 6 hrs. at 65°; after the initial reaction subsides there is obtained 42% 5-nitro-3-benzyltetrahydro-1,3-oxazine (II), m. 143-4°. To 7.5 g. I and 4 g. NaOH in 20 H_2O is slowly added 39 ml. 30% NaOH at 20°, the mixt. stirred for 1 hr., decanted, with HOAc , acid. with Et_2O , and exapd. Treatment of the oily residue with aq. HCl gives 2 g. 5-nitro-3-benzyltetrahydro-1,3-oxazine·HCl, m. 219-12°. I (1 g.) reduced with 3.5 moles H in the presence of Pd gives the 5-amino deriv. Chester Haeck

URBAN II, T.; GURNE, A.

"Reactions of Aliphatic Nitro Compounds. XI. A Few Derivatives of Tetrahydroxazine with Nitromethane, Formaldehyde, and Benzylamine", p. 175, (RODZNIKI CHEMII, Vol. 20, No. 2, 1977, Warsaw, Poland)

CO: Monthly List of East European Acquisitions (FEAL), LC, Vol. 4, No. 3, March 1955, Uncl.

✓ Reactions of cyanoguanidine with aromatic amines. V.
Preparation of new derivatives of 1-phenyl-3-emidinylureas,
their transformation into diaryl urea derivatives. ¹Adriusz
Urbanski, Barbara Skowronska-Serafinowa, and Helena
Dolinska (Inst. Technol., Warsaw). *Roczniki Chem.*
28, 423-37 (1954) (English summary); cf. C.A. 49, 863f.
The following $p\text{-RC}_6\text{H}_4\text{NHCONHC(NH}_2)_2\text{NH}$ were ob-
tained: R = Cl (I), m. 143-4°; R = SO₃H (II), m. 267-9°;
R = NH₂ (III), m. > 300° (hydrochloride). Boiling of I
with PhNH₂ gives $p\text{-ClC}_6\text{H}_4\text{NHCONHC}_6\text{H}_5$, but on pro-
longed boiling I yields p,p' -dichlorocarbonylurea. The cor-
responding unsym. carbonylureas from II and III could not
be obtained. I shows a strong bacteriostatic action against
saprophytic mycobacteria. R. Dowbenko

② MSF

URBANSKI, T.

POL.

New thiosemicarbazones, T. Urbanski and Cz. Relicki-
(Inst. Technol., Warsaw). Recent Chem. 28, 677-8
(1954) (English summary). Thiosemicarbazones of the
following acids were prepd.: p-acetamidobenzoylformic, m.
190°; p-hydroxybenzoylpropionic, m. 192°; p-acetamido-
benzoylpyruvic, m. 178-80°; and of the *Et* esters of the
following substituted acetic acids: nitrobenzoyl, m.
108°; p-aminobenzoyl, m. 145°; nicotinoyl, m. 217°;
isonicotinoyl, m. 102°; and p-acetamidobenzoylpyruvic
acid, m. 125°. No preparative details given. The compds.
are being tested for tuberculostatic activity. C. P.

Urbanaki, T.: Teoria nitrowania. Warszawa: Państwowe
Wyd. Naukowe. 1955. 130 pp. zł. 18 50.

Chem

Urbanaki, T. The theory of nitration.

URBANSKI Y.

3650

547.232 : 847.722.5 : 841.572 : 545.823

Urbański T., Ceclerska D. On Aliphatic Nitrocompounds. The Structure of some Aliphatic and Heterocyclic Nitrocompounds on the Basis of Ultraviolet Absorption Spectrum. CH

„O nitrozwiązkach alifatycznych. Budowa niektórych nitrozwiązków alifatycznych i heterocyklicznych na podstawie badań widma absorpcji w ultrafiolecie”. Roczniki Chemii (PAN). No. 1, 1955, pp. 11—21, 5 figs., 1 tab.

The authors examined the ultraviolet adsorption spectra of 20 aliphatic and heterocyclic nitrocompounds, nitroparaffins and their derivatives, finding that certain nitrocompounds do not show the maximum of absorption in the proximity of $\lambda = 370 \text{ m}\mu$, which is characteristic for the nitro-group. The authors explain this by the formation of chelate rings through hydrogen bonds between both oxygen atoms of the nitro-group and the hydrogen atoms of at least two hydroxyl groups, or by

the formation of hydrogen bonds between one of the oxygen atoms of the nitro-group and the hydrogen of the amino-group.

MA
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URBANSKI, T.

Syntheses and degradation of some derivatives of tetrahydro-1,3-oxazines. D. Gurne and T. Urbanski (Polish Acad. Sci., Warsaw). *Bull. acad. polon. sci. Classe III, 3*, 175-8 (1955) (in English); cf. C.A. 42, 1756. RCH_2NO_2 with CH_3O and PhCH_2NH_2 gave the following 5-nitro-5-alkyl-3-benzyltetrahydro-1,3-oxazines (I) (alkyl and m.p. given): Me (II), 66-8°; Et (III), 68-70°; Pr (IV), 46-8°; I (R = CH_2OH) (V), m. 140-2° (C.A. 49, 8326), with NaOMe gave I (R = H) (VI), m. 44-6°. I heated 5 hrs. with concd. HCl under ultraviolet light or preferably with

1% HCl-EtOH gave the following $\text{HOCH}_2\text{C}(\text{NO}_2)\text{RNIICH}_2\text{Ph.HCl}$ (VII) (R and m.p. given): Me, 102-4° (N,O-di-Bz deriv., m. 112-14°); Et, 150-2° (N,O-di-Bz deriv., m. 105-7°); Pr, 138-8° (N,O-di-Bz deriv., m. 90-2°); HOCH_2 , 177° (decomps.) (N,O-tri-Ac deriv., m. 93-100°; H, 150° (decomp.). VII warmed with CH_3O gave I. VII with MeONa gave the Na salts which with CO_2 gave the following $\text{O}_2\text{NCH}_2\text{RCH}_2\text{NIICH}_2\text{Ph}$ (R, m.p. of HCl salt, and m.p. of N-tosyl deriv. given): Me, 148-50°, 82-4°; Et, 150-1°, 83-0°; Pr, 153-4°, 110-12°; H, 147° (decomp.). (N-Ac deriv., m. 103-5°). The structures were established by the analytical results, by formation of oily N-nitroso compds. which warmed with HCl gave the amines, and through the Bz, Ac, and tosyl derivs. J. E. A.

URBANSKI, T

✓ Same properties of tetrahydro-1,3-oxazines derived from 1-nitrobutane or 1-nitroisobutane. T. Urbanski, J. Kolesińska, and H. Piotrowska (Polish Acad. Sci., Warsaw). *Bull. Acad. Polon. Sci., Classe III*, 3, 179-82 (1955) (in English); cf. C.A. 49, 13998i.—PrNO₂ (C.A. 42, 1756), EtNO₂ [Roczniki Chemii 26, 182 (1952)], and MeNO₂ (C.A. 46, 7993c) react with CH₃O and NH₃ to form several ring compds. whose formation must be due to a reactive N—H group. But BuNO₂ or iso-BuNO₂ (1 mole) heated several hrs. on the steam bath with 3 moles CH₃O and 1 mole NH₃, dried and dissolved in alc.-HCl gave only 5-nitro-5-propyl-tetrahydro-1,3-oxazine-HCl (I), m. 180-2° (picrate, m. 163-4°), or its 5-iso-Pr isomer-HCl (II), m. 190° (picrate, m. 167-8°), and HOCH₂C(NO₂)PrCH₂NH₂ (III), m. 169-70° (HCl salt) (O,N-dibenzoyl deriv., m. 101-2°), or its iso-Pr isomer (IV), m. 165-7° (HCl salt) (picrate, m. 158-60°). All attempts to combine further I and II with CH₃O failed, indicating low activity of the N—H group. I and II were also prepd. by heating 2-nitro-2-propyl-1,3-propanediol or the iso-Pr isomer with 1 mole CH₃O and 1 mole NH₃. I and II gave oily nitroso derivs. which, warmed with HCl gave I and II. The free bases I and II gave the corresponding methiodides, m. 190-200°, and 201-2°, resp. I and II boiled several hrs. with concd. HCl gave III and IV, resp. The free bases III and IV with CH₃O gave the bases I and II, resp. The bacteriostatic concn. of I against various *Mycobacteria* is 62.5-125 mg. % (cf. C.A. 47, 1017). J. Aust. B. Austin

CHIMIA NSH, 1.

Poland/ Organic Chemistry - Synthetic organic chemistry

E-2

Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11781

Author : Piotrowska H., Urbanski T.

Inst : Polish Academy of Sciences

Title : On Derivatives of 5-Nitrotetrahydro-1,3-Oxazine Substituted in Position 2.

Orig Pub : On the formation of derivatives of 5-nitrotetrahydro-1,3-oxazine substituted in position 2.

Bull. Acad. polon. sci., 1955, Cl.3.3, No 7, 389-390 (English)

Abstract : See RZhKhim, 1956, 39594.

Card 1/1

URBANSKI, T.

✓ Anti-tubercular properties of some derivatives of 1 : 3-benzoxazine.
T. Urbanski, D. Gürno, Z. Eckstein, and S. Slopek (*Bull. Acad.
polon. Sci.*, III, 1955, 3, 397-399).—A number of benz-1 : 3-
oxazine derivatives were prepared and shown to be bacteriostatic.
The bacteriostatic concentrations of some of the compounds *in
vivo* against saprophytic *Mycobacteria* are given, the 6-bromo-3-hexyl-
and -3-benzyl-3 : 4-dihydro-deriv. being very effective although
they have LD₅₀ 3 g./kg. per os.

R. J. MAGEE.

CH (3)

URBANSKI, J.

5

✓ Reactions of 5-nitro-1,3-dioxane with diazo compounds and the synthesis of aryldiazotized dials. Z. Eckstein and T. Urbanski. *Bull. acad. polon. sci., Classe III*, 3, 433-6 (1953); St. Gochenour and Degering, *C.A.* 43, 4646; U.S. 2,474,779 (C.A. 43, 8153i); U.S. 2,474,780 (C.A. 43, 8154a).--Derivs. of 5-nitro- (I) and 5-hydroxymethyl-m-dioxane (II) with diazo compds. formed azo compds. when the pH was maintained between 7.5-8.5 with KOH. II treated with bare lost a mol. of CH_2O and the resulting azo compds.

Chem

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$\text{R}^1\text{R}^2\text{C}(\text{O})\text{CHR}^3\text{C}(\text{NO}_2\text{R}^4\text{N}:\text{NAr})\text{CHR}^5\text{O}$ (III) were identical with those formed by I. The following III were obtained ($\text{R}^1, \text{R}^2, \text{R}^3, \text{Ar}$, m.p., % yield): II, Ph, H, Ph, 107-8.5°, 59.1; H, Ph, H, p-ClC₆H₄, cis-(IV), 111.5-113° 2'.0, and trans-IV, 165-7° (decompn.), 7.2; H, Ph, H, p-O₂NC₆H₄, 140-8° (decompn.), 27.8; H, Ph, H, p-MeC₆H₄, 125.5-27°, 58.2; H, Ph, H, 2-C₆H₅, cis-(V), 126.5-8.0°, 18.2, and trans-V, 182-3° (decompn.), 5.6; H, Ph, Me, Ph, 145-0°, 40.9; H, Ph, Me, p-ClC₆H₄, 155.5-0.5°, 66.4; H,

1/2

2. Eckstein...

Ph, Me, *p*-O₂N₂C₆H₄, 136-7° (decompn.), 52.4; H, Ph, Me, *p*-MeC₆H₄, 111-2°, 31.1; H, Ph, Me, 2-C₆H₅, 112-3° 31.9; H, *p*-ClC₆H₄, Me, *p*-O₂N₂C₆H₄, 187-8° (decompn.), 28.0; H, Ph, Pr, *p*-ONC₆H₄, 137-8.5°, 63.4; H, Ph, *iso*-Bu, *p*-O₂NC₆H₄, 121.5-8.6°, 18.5; Me, Me, Me, Ph, 99-100.5°, 20.5; Me, Me, Me, *p*-ClC₆H₄, 138.5-10°, 38.1; Me, Me, Me, *p*-O₂N₂C₆H₄, *cis*-(VI), 129-1°, 11.7, *trans*-VI, 163-4°, 29.6; Me, Me, Me, 2-C₆H₅, 112-14°, 21.0; Me, Me, H, Ph, 94-5°, 86.8; Me, Me, H, *p*-ClC₆H₄, 142-3.5° (decompn.), 91.8; Me, Me, H, *p*-O₂NC₆H₄, 173-4° (decompn.), 72.5; Me, Me, H, *p*-MeC₆H₄, 124.5-20°, 91.2; Me, Me, H, 2-C₆H₅, 133-4°, 86.4; Me, Me, *p*-ONC₆H₄, 168-9° (decompn.), 73.5. The isomers of IV and V were sepd. by fractional crystn. and on hydrolysis gave the same ar/kizonitro diols. They also gave the same ultraviolet spectra. The following ultraviolet spectra data were obtained (max., ε): *cis*-IV 291, 15,810, 405-6, 320; *trans*-IV 291-6, 12,260, 405, 261.4; *cis*-VI 286-8, 19,630, 430-5, 248.5; *trans*-VI 285, 18,010, 430-5, 233.5. Hydrolysis of III with alc. HCl gave the corresponding ketone or aldehyde, as well as ArN:NC(CH₂OH)₂NO₂ (VII). The following derivs. of VII were obtained (Ar, m.p., % yield): Ph, 97-9°, 82.0; *p*-ClC₆H₄, 97-8°, 72.0; *p*-O₂NC₆H₄, 114-16°, 73.6; *p*-MeC₆H₄, 95-7°, 65.6; and 2-C₆H₅, 107-8°, 63.9. VII warmed with BzH and the H₂O removed azetropically yielded cyclic acetals.

Francis Taylor, Jr.

2/2

FM
JSH

URBANSKI, T.

Eckstein, Z.; Urbanski, T. On the alkylation of derivatives of 5-nitro-1,3-dioxane.
In English. p. 489.

MATEMATYKA

Vol. 3, No. 9, 1955

Warszawa, Poland

SO: Monthly List of East European Accessions, (EFAL), LC, Vol. 5, No. 10
Oct. 56

URBANSKI, T.

Urbanski, T. On the productions of the reaction of 1-nitropropane with formaldehyde and ethylenediamine. In English. p. 493.

MATEMATYKA

Vol 3, No. 9, 1955 Warszawa, Poland

SOURCE: EEAL, LC, Vol. 5, No. 10 Oct. 1956

URBANSKI, T.

Poland/Chemistry of High-Molecular Substances, F

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61691

Author: Urbanski, T.

Institution: None

Title: Conference on Macromolecular Chemistry at Zuerich

Original

Periodical: Symposium z zakresu chemii makromolekularnej w zurychu, Wiadom chem., 1955, 9, No 12, 662-667; Polish

Abstract: None

Card 1/1

URBAŃSKI, T.

"Chemia i technologia materiałów wybuchowych" (Chemistry and technology of explosive materials), by T. Urbański. Reported in New Books (Nowe Książki), No. 14, July 15, 1955

URBANSKI, T.

Skowronska-Serapinowa, B.; Dabrowski, H. Reactions of cyanoguanidine with aromatic amines. VI. Some new derivatives of amidine-phenyl-urea and their reactions with aniline. p. 450.
ROCZNIKI CHEMII, Warszawa, Vol. 29, no. 2/3, 1955.

SO: Monthly List of East European Accessions, (SEAL), LC, Vol. 4, no. 10, Oct. 1955, Uncl.

LIRBAWSKI, T.

expresses why...
instead of OH group. U. and C. suppose that the cause is
more strong electron repelling character of the amine group.
A. Semakova

URBANSKI, Tadeusz

10

Reactions of aliphatic nitro compds. XIII. Liebermann reactions of secondary amines containing a nitro group. Tadeusz Urbanski and Zdzisław Eckstein (Inst. Technol., Warsaw). *Kocinski Chem.* 29, 916-18 (1955) (English summary); cf. *C.A.* 49, 11414d. The Liebermann test was modified for secondary amines contg. nitro groups, which were found to interfere under the usual conditions. The soln. of nitrosamine in H_2SO_4 with phenol was not warmed. XIV. Action of nitroparaffins on reaction between 2-aminopyridine and formaldehyde; Tadeusz Urbanski and Barbara Słowczyńska-Szanińska. *Ibid.* 367-74. $HCHO$ (I), 2-aminopyridine (II), and 1-nitropropane stirred together at $20-30^\circ$ and allowed to stand overnight gave 5-nitro-6-ethyl-1,3-dioxane, m. $52-4^\circ$, and N,N' -di(2-pyridyl)-methylene diamine (III), m. $130-1^\circ$ (methoxide, m. $225-8^\circ$), gives Liebermann test for secondary amine. III was also obtained by mixing nitro alcs. (2-ethyl-2-nitro-1,3-propanediol, 2-methyl-2-nitro-1,3-propanediol, and 2-methyl-2-nitropropanol) with II. The catalytic effect of primary and secondary nitroparaffins, $PhNO_2$, $CE_2(CO_2Et)_2$, HCl , $PhOH$, NH_4Cl , and β -hydroxypyridine on the reaction of I and II to give III was also studied. XV. Interpretation of ultraviolet absorption spectra of nitroparaffin derivatives. Tadeusz Urbanski. *Ibid.* 375-8. On the basis of ultraviolet spectra (near 270 m μ) of some aliphatic amino nitro compds., a suggestion based on analogy with amino acids was made that the H of an amino group can be bound with

both O atoms of a nitro group by means of 2 H bonds. XVI. Products of reaction of 1-nitro-n-butane with formaldehyde and ammonia. Tadeusz Urbanski and Hanna Płotrowski. *Ibid.* 379-91. Mixing $BuNO_2$ (I), $HCHO$, and NH_3 in a molar proportion 1:3:1 gave after fractional crystal. from $EtOH$ 5-nitro-5-propyltetrahydro-1,3-dioxane-HCl (II), m. $199-2^\circ$ (dimethiodide, m. $199-200^\circ$), and 2-nitro-2-(hydroxymethyl)pentylamine-HCl (III), m. $169-70^\circ$ (O,N-di-Bz deriv., m. $101-2^\circ$). Use of 2-nitro-2-propyl-1,3-propanediol (IV) instead of I gave higher yields of II and III. I treated with 3 moles $HCHO$ in excess NH_3 at room temp. gave 5-nitro-5-propylhexahydropyrimidine-1HCl (V), m. $171-3^\circ$ (di-N-nitroso deriv., m. $99-100^\circ$). V warmed with aq. $EtOH$ gave 2-nitro-2-propyl-1,3-propylene diamine, m. $178-9^\circ$ (di(p-nitrobenzoyl) deriv., m. $216-17^\circ$). Using IV instead of I gave a better yield of V. IV warmed with excess NH_3 gave 3,7-dinitro-3,7-dipropyl-1,5-diazacyclooctane (VI), m. $73-4^\circ$ (mono-HCl salt, m. $173-4^\circ$; mono-N-nitroso deriv., m. $110-11^\circ$). VI.HCl was of moderate bacteriostatic activity *in vitro* against saprophytic mycobacteria. XVII. Products of reaction of 1-nitroisobutane with formaldehyde and ammonia. Tadeusz Urbanski and Janina Kolesińska. *Ibid.* 392-8. $Me_2CHCH_2NO_2$ (I) (1 mole) treated with 3 moles $HCHO$, and 1 mole NH_3 gave after fractional crystal. of their HCl salts from $EtOH$ 5-nitro-5-isopropyltetrahydroxazine (II).HCl, m. 190° [yield

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7

(7)

TADEUSZ URBANISKI

2/12

10%; picrate, m. 167-8° (decolorn.); N-Ms MeI salt, m. 201-2°, and 2-nitro-2-(hydroxymethyl)isopentylamine (III). HCl, m. 165-7° (yield 6%; picrate, m. 158-60°). II and III were also prepd. by treating 1 mole 2-nitro-2-isopropyl-1,3-propanediol (m. 84-6°, obtained by heating I with HCHO at pH 7.5) with 1 mole HCHO, and 1 mole NH₄OH. II warmed on a steam bath with concd. HCl 6 hrs. gave HCHO and III.HCl, while III warmed with HCHO gave isovaleraldehyde. Tadeusz Urbaniski, Zygmunt Eckstein, and Wiesław Sabótka. *Ibid.* 399-409.—The residue from the prepn. of 1-nitro-4-methyl-2-pentanol (I) according to the method of Henry [*Rec. trav. chim.* 16, 201(1897)], Mousset [*Rec. trav. chim.* 21, 65(1902)], or Bouveault and Wahl [*Bull. soc. chim. France* 29, 643(1903)] was found to contain 5-nitro-2,8-dimethyl-4,6-nonanediol (II), m. 92-3° (from CCl₄); diacetate, m. 108.5°. A yield of 6.5 g. II was also obtained when 29.4 g. I was treated with 20 g. isovaleraldehyde (III) in the presence of 1.6 ml. NEt₃. Chlorination and bromination of the Na salt of I led to 1-chloro-1-nitro-4-methyl-2-pentanol (IV), b_p 93.5-9.0°, and 1-bromo-1-nitro-4-methyl-2-pentanol (V), b_p 107-3°, resp.

Treatment of IV and V with excess III led to 5-chloro-5-nitro-2,8-dimethyl-4,6-nonanediol, m. 123-4° (from CCl₄), and 5-bromo-5-nitro-2,8-dimethyl-4,6-nonanediol (VI), m. 133-4° (from CCl₄-CHCl₃), resp. II reacted with aldehydes, yielding 2-R-substituted 5-nitro-4,6-diisobutyl-1,3-dioxanes; when R = Ph, p-ClC₆H₄, p-MeOC₆H₄, p-O₂N-C₆H₄, the m.p. was 140, 160, 148, 176°, resp. Similarly, VI reacted with benzaldehyde to give 2-phenyl-5-bromo-5-nitro-4,6-diisobutyl-1,3-dioxane, m. 153°. IV reacted with HCHO in the presence of triethylamine to give 2-chloro-2-nitro-5-methyl-1,3-hexanediol, m. 103.6-4.0° (from CCl₄ and then C₆H₆). XIX. Preparation of alkyl resins from nitrophthalic acids and ethylene glycol. Tadeusz Urbaniski and Marceli Ficines. *Ibid.* 412-15.—4-Nitrophthalic acid was esterified by (HOCH₂)₂ at 150-8° yielding a resin more readily than 3-nitrophthalic acid (I). Formation of a seven-membered ring by means of a hydrogen bond between the nitro group and the carboxyl group of I was suggested as an explanation.

P. Dreyfus

8/24

URBANSKI, T.:

POLAND

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UREANSKI, T.

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